

**FIELD MODIFICATION FORM
LOWER PASSAIC RIVER RESTORATION PROJECT**

Date: January 8, 2007

Document: Final QAPP, August 2005

Activity: Analysis of High Resolution Sediment Core Samples Collected from Dundee Lake

Requested Modifications: In January 2007, Malcolm Pirnie will collect 8-10 high resolution sediment cores from Dundee Lake. Following sediment core processing, the resultant sediment samples will be analyzed for Polychlorinated Biphenyl (PCB) Congeners, Dioxins/Furans, Pesticides, Polycyclic Aromatic Hydrocarbons (PAHs), Target Analyte List (TAL) metals including mercury plus titanium, and radiochemistry (Cs-137 on all sediment segments plus Be-7 on the surface sediment slices). If sufficient sample volume is available, the sediments will also be analyzed for total organic carbon (TOC) and particle size distribution by laser light scattering (ASTM D4464).

Three sediment cores will be selected by Dr. Edward Garvey and the field staff from the 8-10 candidate cores based on depth recovered, percent recovery, absence of voids, and other visually-determined core parameters. Each of the selected 3-inch inside diameter cores will be divided into approximately 30 individual sediment segments. One aliquot from each sediment segment will be shipped to Axys Analytical for organic analyses (PCBs, Dioxins/Furans, Pesticides, and PAHs) and a second aliquot of each segment will be shipped to Severn Trent Laboratories (STL) for radiochemistry, TAL metals plus titanium, TOC, and particle size distribution. A co-located 2 inch diameter core will also be collected, sampled, and tested for Grain Size by ASTM D422. If a distinct petroleum product layer (such as coal tar material) is visually detected while processing the cores, a portion will be collected and submitted for total petroleum hydrocarbon (TPH) analysis by the NJDEP method specified in Attachment 12 of the August 2005 QAPP. A portion will also be archived for possible further characterization.

The laboratories (Axys Analytical and STL) will homogenize the samples they receive prior to sub-sampling for analyses. A portion of the sediment sample homogenized by STL will be submitted for radiochemistry dating. The following modifications to the QAPP of August 2005 are requested for the management and analyses of the Dundee Lake sediment samples:

1. To improve the analytical resolution for pesticides, Axys Analytical will determine the pesticide parameters following their high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) method entitled, "Analytical Procedures for Organochlorine Pesticides by Isotope Dilution HRGC/HRMS", MLA-028 Rev. 02, 03-August 2006. It is requested that the HRGC/HRMS method be utilized for the Dundee Lake samples and for future Passaic River samples collected for pesticides, since the HRGC/HRMS method will improve the resolution of interfering compounds. A summary of this method is attached. The other organic parameters submitted to Axys Analytical

will be analyzed by methods previously referenced in the Final QAPP.

2. STL Burlington will perform the metals, TOC, and particle distribution analyses on the Dundee Lake sediment samples. This laboratory choice was made due to the limited volume of sediment sample that we anticipate will be available from the finely-segmented, 3-inch sediment cores. STL Burlington is both NELAP (National Environmental Laboratory Accreditation Program) and New Jersey Department of Environmental Protection certified. STL Burlington is also approved to follow the USEPA CLP ILM05.3 Statement of Work for Inorganics. STL's procedures for metals analyses are capable of achieving the low reporting limits specified in the QAPP on the very limited sediment sample volumes that will be available.

3. It is requested that STL Richland be approved as an alternate laboratory (to OutReach Analytical) to perform the radiochemistry analyses on the sediment samples. STL Richland is one of the few commercial radiochemistry laboratories capable of achieving the low detection limits on the limited sample volume that will be available. The laboratory is both NELAP and Army Corps of Engineers certified. If STL Richland is utilized to perform the radiochemistry for the Dundee Dam samples, the limited sediment sample volume available will be conserved for other analyses. STL Burlington will homogenize each sediment sample, remove the small portion required for metals analysis, and subsequently forward a sample aliquot to STL Richland for radiochemistry. If sufficient material is available, STL Burlington will also remove an aliquot for TOC and particle distribution analyses before shipping the remainder to the radiochemistry lab.

4. After processing, the sediment samples for organic parameters (PCBs, Dioxins/Furans, Pesticides, and PAHs) will be stored in a freezer. After they are frozen at least overnight they will be shipped while preserved on dry ice (or alternately using wet ice) to Axys Analytical. Upon arrival at Axys Analytical the samples for the organic parameters will immediately be stored in a freezer. Freezing the samples will allow for the extension of analytical holding times. The following are the holding times for the organic parameters based upon holding times for frozen samples previously specified in the earlier QAPP field modification of October 10, 2005:

- a. For pesticide analyses of sediments and soils the holding time, if frozen and stored in the dark in the laboratory, will be up to a maximum of 299 days prior to extraction.
- b. For PAH analyses of sediments and soils the maximum holding time will be up to 100 days prior to extraction.
- c. The holding times for PCB Congeners and Dioxin/Furans on the sediment samples will be one year from collection if stored frozen and in the dark by the laboratory.

Note: If necessary to ship the sample to the laboratory using wet ice at 4°C (instead of preserved on dry ice), the number of days in shipment will be subtracted from the holding times listed above.

Rationale: The modifications described above will improve the overall quality of the analytical
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Dundee Lake Hi Res Coring

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data, as described below:

1. The low resolution pesticide method employed for Lower Passaic River field investigations conducted during 2005-2006 and listed in the Final QAPP had difficulties quantifying all of the pesticides at the required reporting limits due to high levels of interfering compounds. The Axys Analytical HRGC/HRMS method for pesticides has lower detection limits and will improve the resolution of interfering compounds, therefore improving the quality of the pesticide data.
2. Use of the proposed alternative laboratories (STL Burlington and STL Richland) for the metals and radiochemistry analyses will generate data at the reporting limits specified in the QAPP on the limited volume of sediment sample that we anticipate will be available.
3. Preservation of the samples for organics analyses by freezing immediately after processing, shipment on ice, and continued freezing upon arrival at the laboratory will reduce the need for storage of samples at the field facility, permit the laboratory to more efficiently schedule the analyses, and conserve project budget (additional field effort associated with sending multiple sample shipments to the laboratory based on lab capacity not necessary).

Attachments: Attachments include a summary of the Axys Analytical procedure for pesticide analysis by HRGC/HRMS, as well as copies of the STL procedures for radiochemistry and metals plus mercury.

Attachment 1 - Axys Analytical HRGC/HRMS Pesticide Standard Operating Procedure (SOP) Summary

Attachment 2 - Axys Analytical HRGC/HRMS Pesticide Detection Limits

Attachment 2.1 - Axys Analytical Pesticide HRGC/HRMS QC Specifications


Attachment 3 - STL Richland Gamma Spec SOP


Attachment 4 - STL Burlington Mercury SOP

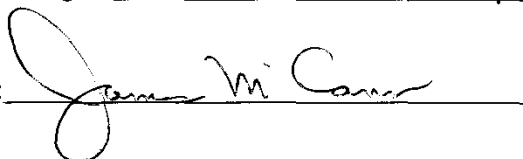
Attachment 5 - STL Burlington Metals by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES)

Attachment 6 - STL Burlington Metals by ICP-Mass Spectroscopy (MS)

Attachment 7 - STL Burlington Particle-Size Distribution by Laser Light Scattering (ASTM D4464)

Malcolm Pirnie Project Manager: 

Malcolm Pirnie Deputy Project Manager:  For Scott H. Thompson

Malcolm Pirnie Site QC Officer: 

ATTACHMENT 1

Axys Analytical HRGC/HRMS Pesticide SOP Summary

DETERMINATION OF ORGANOCHLORINE PESTICIDES BY HRGC/HRMS

Extraction and Cleanup Procedures

This method is applicable to the analysis of solids, tissues (including milk and blood), aqueous samples, XAD columns, air samples and solvent extracts. Samples are spiked with a suite of isotopically labelled surrogate standards prior to analysis. Samples are solvent extracted. The extracts are cleaned up and separated into two fractions, E1 and E2, using Florisil. Each fraction is spiked with isotopically labelled recovery (internal) standard(s) prior to analysis for a suite of organochlorine pesticides by high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

HRGC/HRMS Analysis

Analysis of the E1 and E2 fractions is performed on a high-resolution mass spectrometer (HRMS) coupled to a high-resolution gas chromatograph (HRGC) equipped with a J&W DB-5 chromatography column (60 m, 0.25 mm i.d., 0.10 µm film thickness). The HRMS is operated at a static (8000) mass resolution (10% valley) in the electron ionization (EI) mode using multiple ion detection (MID). The ions acquired are presented in Tables 1 and 2.

Initial calibration is performed using a multi-point calibration series of solutions that encompass the working concentration range. Calibration is verified at least once every twelve hours by analysis of a mid-level calibration solution

Analyte Identification

A chromatographic peak is identified as a target compound if the following criteria are met for the quantification and confirmation ions (where confirmation ions are available):

1. Peak response must be at least three times the background noise level.
2. The peak retention time must be within the window predicted from the initial calibration runs and the surrogate standard retention times.
3. Peak maxima for quantification and confirmation ions must coincide within two seconds.
4. The relative ion abundance ratios must be within 20% of the theoretical except for oxychlordane and labelled methoxychlor that must be 30% and 50% respectively.

Quantification Procedures

Target concentrations are determined with respect to labelled surrogate standards as shown in Tables 1 and 2. Mean relative response factors (RRF) determined from the initial calibration runs are used to convert raw peak areas in sample chromatograms to final concentrations as follows:

$$\text{Concentration of Target} = \left(\frac{\text{area of Target}}{\text{area of Surrogate}} \right) \times \left(\frac{\text{weight of Surrogate}}{\text{RRF}} \right) \times \left(\frac{1}{\text{weight of sample}} \right)$$

$$\text{where RRF} = \left(\frac{\text{area of Target}}{\text{area of Surrogate}} \right) \times \left(\frac{\text{concentration of Surrogate}}{\text{concentration of Target}} \right)$$

Final concentrations are recovery corrected by the method of quantification.

Sample specific detection limits (SDLs), reported with the analytical results, are determined from the analysis data by converting the minimum detectable signal to a concentration following the same procedures used to convert target peak responses to concentrations. The estimated minimum detectable area is determined as three times the height of the noise in the m/z channel of interest, converted to an area using the area height ratio of the corresponding labelled surrogate peak.

Recoveries of surrogates are determined similarly against the recovery (internal) standard and are used as general indicators of overall analytical quality.

QA/QC

Samples are analyzed in batches consisting of a maximum of twenty samples, a procedural blank and a spiked matrix (OPR) sample. Sample duplicates or matrix spike/matrix spike duplicate (MS/MSD) pairs may be analyzed on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria presented on the analysis reports.

All aspects of the method are described in detail in AXYS' document MLA-028 "Organochlorine Pesticides by Isotope Dilution HRGC/HRMS".

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Table 1. Analyte Ions Monitored, Surrogates Used, and RRF Determination for E1 Pesticides by HRGC/HRMS

(No entry in the "RRF Used" field designates an RRF derived from that same compound.)

Analyte Name	Nominal Amt Spiked (ng)	Quantified against labelled standard	RRF Determination	mass1	mass2	m1/m2 ratio	Ion Ratio Tolerance (+/- %)
1,3/1,4-Cl ₂ -BZ		¹³ C ₆ -1,4-Cl ₂ -BZ		145.969	147.966	1.56	20
1,2-Cl ₂ -BZ		¹³ C ₆ -1,4-Cl ₂ -BZ		145.969	147.966	1.56	20
1,3,5-Cl ₃ -BZ		¹³ C ₆ -1,2,3-Cl ₃ -BZ		179.93	181.927	1.03	20
1,2,4-Cl ₃ -BZ		¹³ C ₆ -1,2,3-Cl ₃ -BZ		179.93	181.927	1.03	20
1,2,3-Cl ₃ -BZ		¹³ C ₆ -1,2,3-Cl ₃ -BZ		179.93	181.927	1.03	20
1,2,4,5/3,5-Cl ₄ -BZ		¹³ C ₆ -1,2,3,4-Cl ₄ -BZ		213.891	215.888	0.77	20
1,2,3,4-Cl ₄ -BZ		¹³ C ₆ -1,2,3,4-Cl ₄ -BZ		213.891	215.888	0.77	20
Cl ₅ -BZ		¹³ C ₆ -Cl ₅ -BZ		247.852	249.849	0.62	20
HCB		¹³ C ₆ -HCB		283.81	285.807	1.25	20
TCMX		¹³ C ₆ -HCB		241.922	243.919	0.78	20
Cl ₆ -Butadiene		¹³ C ₆ -1,2,3,4-Cl ₄ -BZ	1,2,3,4-Cl ₄ -BZ	189.872	187.875	1.32	20
alpha-HCH		¹³ C ₆ -gamma-HCH		216.915	218.912	0.77	20
beta-HCH		¹³ C ₆ -beta-HCH		216.915	218.912	0.77	20
gamma-HCH		¹³ C ₆ -gamma-HCH		216.915	218.912	0.77	20
delta-HCH ¹		¹³ C ₆ -delta-HCH		216.915	218.912	0.77	20
Heptachlor		¹³ C ₁₀ -Heptachlor		271.81	273.81	1.24	20
Aldrin		¹³ C ₁₂ -Aldrin		262.857	264.854	1.55	20
Octachlorostyrene		¹³ C ₁₀ -trans-Chlordane		270.844	272.841	0.62	20
Oxychlordane		¹³ C ₁₀ -Oxychlordane		262.857	264.854	1.55	20
trans-Chlordane		¹³ C ₁₀ -trans-Chlordane		271.81	273.807	1.24	20
cis-Chlordane		¹³ C ₁₀ -trans-Chlordane		271.81	273.807	1.24	20
o,p-DDE		¹³ C ₁₂ -p,p-DDE		246	247.997	1.56	20
p,p-DDE		¹³ C ₁₂ -p,p-DDE		246	247.997	1.56	20
trans-Nonachlor		¹³ C ₁₀ -trans-Nonachlor		271.81	273.807	1.24	20
cis-Nonachlor		¹³ C ₁₀ -cis-Nonachlor		271.81	273.807	1.24	20
o,p-DDD		¹³ C ₁₂ -o,p-DDT		235.008	237.005	1.56	20
p,p-DDD		¹³ C ₁₂ -o,p-DDT		235.008	237.005	1.56	20
o,p-DDT		¹³ C ₁₂ -o,p-DDT		235.008	237.005	1.56	20
p,p-DDT		¹³ C ₁₂ -p,p-DDT		235.008	237.005	1.56	20
Photomirex		¹³ C ₁₀ -Mirex	Mirex	269.813	271.81	0.52	20
Mirex		¹³ C ₁₀ -Mirex		269.813	271.81	0.52	20

¹ delta-HCH normally will elute primarily in the E2 fraction and can be quantified solely from this fraction. Recoveries of ¹³C-delta-HCH may be reported as the sum of the E1 and E2 recoveries if significant concentrations of ¹³C-delta-HCH are observed in the E1 fraction.

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Table 1 (Cont'd)

Analyte Name	Nominal Amt Spiked (ng)	Quantified against labelled standard	RRF Determination	mass1	mass2	m1/m2 ratio	Ion Ratio Tolerance (+/-)
Surrogate Standards							
¹³ C ₆ -1,4-Cl ₂ -BZ	8	¹³ C ₁₂ -PCB-52		151.989	153.986	1.56	20
¹³ C ₆ -1,2,3-Cl ₃ -BZ	8	¹³ C ₁₂ -PCB-52		185.95	187.947	1.03	20
¹³ C ₆ -1,2,3,4-Cl ₄ -BZ	8	¹³ C ₁₂ -PCB-52		221.908	223.905	2.08	20
¹³ C ₆ -Cl ₅ -BZ	8	¹³ C ₁₂ -PCB-52		255.869	257.866	1.55	20
¹³ C ₆ -HCB	8	¹³ C ₁₂ -PCB-52		289.83	291.828	1.25	20
¹³ C ₆ -beta-HCH	8	¹³ C ₁₂ -PCB-52		222.935	224.932	0.77	20
¹³ C ₆ -gamma-HCH	8	¹³ C ₁₂ -PCB-52		222.935	224.932	0.77	20
¹³ C ₆ -delta-HCH	8	¹³ C ₁₂ -PCB-52		222.935	224.932	0.77	20
¹³ C ₁₀ -Heptachlor	8	¹³ C ₁₂ -PCB-138		276.827	278.824	1.24	20
¹³ C ₁₀ -Aldrin	8	¹³ C ₁₂ -PCB-138		269.88	271.877	1.55	20
¹³ C ₁₀ -Oxychlordane	8	¹³ C ₁₂ -PCB-138		269.88	271.877	1.55	30
¹³ C ₁₀ -trans-Chlordane	8	¹³ C ₁₂ -PCB-138		276.827	278.824	1.24	20
¹³ C ₁₀ -trans-Nonachlor	8	¹³ C ₁₂ -PCB-138		276.827	278.824	1.24	20
¹³ C ₁₀ -cis-Nonachlor	8	¹³ C ₁₂ -PCB-138		276.827	278.824	1.24	20
¹³ C ₁₂ -o,p-DDE	8	¹³ C ₁₂ -PCB-138		258.041	260.038	1.56	20
¹³ C ₁₂ -p,p-DDE	8	¹³ C ₁₂ -PCB-138		258.041	260.038	1.56	20
¹³ C ₁₂ -o,p-DDT	8	¹³ C ₁₂ -PCB-138		247.048	249.045	1.56	20
¹³ C ₁₂ -p,p-DDT	8	¹³ C ₁₂ -PCB-138		247.048	249.045	1.56	20
¹³ C ₁₀ -Mirex	8	¹³ C ₁₂ -PCB-138		276.827	278.824	1.25	20
Recovery Standards							
¹³ C ₁₂ -PCB-52	10			301.963	303.96	0.77	20
¹³ C ₁₂ -PCB-138	10			299.947	301.944	0.77	20

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Table 2. Analyte Ions Monitored, Surrogates Used and RRF Determination for E2 Pesticides by HRGC/HRMS

(No entry in the "RRF Used" field designates an RRF derived from that same compound.)

Analyte Name	Nominal Amt Spiked (ng)	Quantified against labelled standard	RRF Determination	mass1	mass2	m1/m2 ratio	Ion Ratio Tolerance (+/- %)
delta-HCH		¹³ C ₆ -gamma-HCH		216.915	220.909	1.63	20
Heptachlor epoxide		¹³ C ₉ -Heptachlor-Epoxide		354.841	352.844	0.8	20
alpha-Endosulphan		¹³ C ₉ -alpha-endosulphan		264.854	262.857	0.64	20
Dieldrin		¹³ C ₁₂ -Dieldrin		264.854	262.857	0.64	20
Endrin		¹³ C ₁₂ -Endrin		264.854	262.857	0.64	20
beta-Endosulphan		¹³ C ₉ -beta-Endosulphan		264.854	262.857	0.64	20
Endosulphan sulphate		¹³ C ₉ -beta-Endosulphan		264.854	262.857	0.64	20
Endrin aldehyde		¹³ C ₁₂ -Endrin		346.896	344.899	0.64	20
Endrin ketone		¹³ C ₁₂ -Endrin		318.901	316.904	0.64	20
Methoxychlor		¹³ C ₁₂ -Methoxychlor		228.111	227.107	0.17	20
Labelled Surrogates							
¹³ C ₆ -gamma-HCH	8	¹³ C ₁₂ -PCB-153		222.935	224.932	0.78	20
¹³ C ₉ -Heptachlor-Epoxide	8	¹³ C ₁₂ -PCB-153		364.875	362.878	0.8	20
¹³ C ₉ -alpha-endosulphan	8	¹³ C ₁₂ -PCB-153		271.877	269.88	0.64	20
¹³ C ₁₂ -Dieldrin	8	¹³ C ₁₂ -PCB-153		271.877	269.88	0.64	20
¹³ C ₁₂ -Endrin	8	¹³ C ₁₂ -PCB-153		271.877	269.88	0.64	20
¹³ C ₉ -beta-Endosulphan	8	¹³ C ₁₂ -PCB-153		271.877	269.88	0.64	20
¹³ C ₁₂ -Methoxychlor	8	¹³ C ₁₂ -PCB-153		239.148	240.151	30	50
Recovery Standard							
¹³ C ₁₂ -PCB-153	10			299.947	301.944	0.78	20

ATTACHMENT 2

Axys Analytical HRGC/HRMS Pesticide Detection Limits

AXYS METHOD MLA-028 Rev 01
Method Detection Limit for E1 Pesticides in Solid Samples
October-05

TABLE 1

Axys Method: AXYS METHOD MLA-028 Rev 01
Analysis Type: E1 Pesticides
Instrument Type: High Resolution GC/MS
Matrix Spiked: SOLID
AXYS Work Group: WG17119
GC Column Type: DB-5
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, October 26, 1984; no iteration.

MDL 1 Data Filename:	CL53_295F S:24	SAMPLE ID:	WG17119-103,SPM	ANALYSIS DATE:	9-Oct-05
MDL 2 Data Filename:	CL53_295F S:25	SAMPLE ID:	WG17119-104,SPM	ANALYSIS DATE:	9-Oct-05
MDL 3 Data Filename:	CL53_295F S:26	SAMPLE ID:	WG17119-105,SPM	ANALYSIS DATE:	9-Oct-05
MDL 4 Data Filename:	CL53_295F S:27	SAMPLE ID:	WG17119-106,SPM	ANALYSIS DATE:	9-Oct-05
MDL 5 Data Filename:	CL53_295F S:28	SAMPLE ID:	WG17119-107,SPM	ANALYSIS DATE:	9-Oct-05
MDL 6 Data Filename:	CL53_295F S:29	SAMPLE ID:	WG17119-108,SPM	ANALYSIS DATE:	9-Oct-05
MDL 7 Data Filename:	CL53_295F S:30	SAMPLE ID:	WG17119-109,SPM	ANALYSIS DATE:	9-Oct-05
MDL 8 Data Filename:	CL53_295F S:32	SAMPLE ID:	WG17119-111,SPM	ANALYSIS DATE:	9-Oct-05

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE IN ng/g
Based on 10g sample size

Native Analyte	Spiking Level (ng/g)	Number of Observations	Mean (ng/g)	Standard Deviation	Student's t- Value	Method Detection Limit (ng/g)
HCB	0.08	8	0.08	0.002	2.998	0.008 ¹
TCMX	0.08	8	0.08	0.01	2.998	0.035
alpha-HCH	0.16	8	0.14	0.02	2.998	0.066
beta-HCH	0.16	8	0.14	0.01	2.998	0.026
gamma-HCH	0.16	8	0.14	0.003	2.998	0.009
HEPTACHLOR	0.08	9	0.07	0.004	2.998	0.011
ALDRIN	0.16	8	0.14	0.006	2.998	0.017
CL8-STYRENE	0.08	8	0.08	0.008	2.998	0.023
OXYCHLORDANE	0.16	8	0.16	0.012	2.998	0.035
l-CHLORDANE	0.16	8	0.15	0.006	2.998	0.018
c-CHLORDANE	0.16	8	0.16	0.007	2.998	0.022
l-NONACHLOR	0.16	8	0.16	0.006	2.998	0.019
c-NONACHLOR	0.16	8	0.16	0.007	2.998	0.022
o,p-DDD	0.08	8	0.09	0.006	2.998	0.019
p,p-DDD	0.10	8	0.09	0.008	2.998	0.023
o,p-DDE	0.08	8	0.08	0.006	2.998	0.017
p,p-DDE	0.08	8	0.08	0.002	2.998	0.005
o,p-DDT	0.09	8	0.07	0.003	2.998	0.010
p,p-DDT	0.08	8	0.08	0.005	2.998	0.014
MIREX	0.08	8	0.08	0.003	2.998	0.010

NOTE 1: Estimated maximum MDL assigned as 1/10 spiking level

AXYS METHOD MLA-028 Rev 01
Method Detection Limit for E2 Pesticides in Solid Samples
October-05

TABLE 1

Axys Method: AXYS METHOD MLA-028 Rev 01
Analysis Type: E2 Pesticides
Instrument Type: High Resolution GC/MS
Matrix Spiked: SOLID
AXYS Work Group: WG17119
GC Column Type: DB-5
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, October 26, 1984; no iteration.

MDL 1 Data Filename:	CL53_293D S:28	SAMPLE ID:	WG17119-103,,SPM	ANALYSIS DATE:	7-Oct-05
MDL 2 Data Filename:	CL53_293D S:29	SAMPLE ID:	WG17119-104,,SPM	ANALYSIS DATE:	7-Oct-05
MDL 3 Data Filename:	CL53_293D S:30	SAMPLE ID:	WG17119-105,,SPM	ANALYSIS DATE:	7-Oct-05
MDL 4 Data Filename:	CL53_293D S:31	SAMPLE ID:	WG17119-106,,SPM	ANALYSIS DATE:	7-Oct-05
MDL 5 Data Filename:	CL53_293D S:32	SAMPLE ID:	WG17119-107,,SPM	ANALYSIS DATE:	7-Oct-05
MDL 6 Data Filename:	CL53_293D S:33	SAMPLE ID:	WG17119-108,,SPM	ANALYSIS DATE:	7-Oct-05
MDL 7 Data Filename:	CL53_293D S:34	SAMPLE ID:	WG17119-109,,SPM	ANALYSIS DATE:	7-Oct-05
MDL 8 Data Filename:	CL53_293D S:36	SAMPLE ID:	WG17119-111,,SPM	ANALYSIS DATE:	7-Oct-05

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE IN ng/g
Based on 10 g sample size

Native Analyte	Spiking Level (ng/g)	Number of Observations	Mean (ng/g)	Standard Deviation	Student's t- Value	Method Detection Limit (ng/g)
delta-HCH	0.16	8	0.159	0.003	2.998	0.009
Heptachlor-Epoxyde	0.08	8	0.082	0.003	2.998	0.010
alpha-Endosulphan	0.08	8	0.089	0.005	2.998	0.014
Dieldrin	0.08	8	0.086	0.005	2.998	0.016
Endrin	0.08	8	0.081	0.003	2.998	0.010
beta-Endosulphan	0.08	9	0.095	0.008	2.998	0.025
Endosulphan-Sulphate	0.08	8	0.090	0.013	2.998	0.039
Endrin-Aldehyde	0.08	8	0.187	0.033	2.998	0.100
Endrin-Ketone	0.08	8	0.076	0.020	2.998	0.061
Methoxychlor	0.08	8	0.089	0.005	2.998	0.014

ATTACHMENT 2.1

Axys Analytical Pesticide HRGC/HRMS QC Specifications

HR Pesticides

The quality control limits for duplicate samples, procedural blanks, reference samples, surrogate recoveries, and detection limits are specified in the Table 6 for E1 fraction pesticides and Table 7 for E2 fraction pesticides.

Table 6. QC Criteria for E1 Chlorinated Pesticides

Analyte:	Typical Sample Specific Detection Limits					Procedural Blank Level	Acceptable Matrix Spike
	Solid	Aqueous	Tissue	Pulp	XAD Column		
	ng/g	ng/L	ng/g	ng/g	ng	ng	% Recovery
Dichlorobenzenes	0.1	1.0	0.1	0.05	1.0	<1.0	60-130
Trichlorobenzenes	0.1	1.0	0.1	0.05	1.0	<1.0	60-130
Tetrachlorobenzenes	0.1	1.0	0.1	0.05	1.0	<0.5	60-130
Pentachlorobenzene	0.01	0.1	0.01	0.05	0.1	<0.1	70-130
Hexachlorobenzene	0.01	0.1	0.01	0.05	0.1	<0.1	70-130
alpha-HCH	0.02	0.2	0.02	0.01	0.2	<0.2	70-130
beta-HCH	0.02	0.2	0.02	0.01	0.2	<0.2	70-130
gamma-HCH	0.02	0.2	0.02	0.01	0.2	<0.2	70-130
delta-HCH	0.02	0.2	0.02	0.01	0.2	<0.2	70-130
Heptachlor	0.02	0.2	0.02	0.01	0.2	<0.2	70-130
Aldrin	0.02	0.2	0.02	0.01	0.2	<0.2	70-130
Oxychlordane	0.02	0.2	0.02	0.01	0.2	<0.2	70-130
trans-Chlordane	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
cis-Chlordane	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
o,p'-DDE	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
p,p'-DDE	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
trans-Nonachlor	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
cis-Nonachlor	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
o,p'-DDD	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
p,p'-DDD	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
o,p'-DDT	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
p,p'-DDT	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
Mirex	0.02	0.2	0.02	0.01	0.2	<0.1	70-130
Total Toxaphene	0.05	0.5	0.05	NA	0.5	<0.5	60-150
Typical Sample Size:	10 g	1 L	10 g	20 g	1 Column		
Typical Final Vol, µL *	200	200	200	200	200		

*Extract volumes may range from 20 µL to 200 µL. To avoid excessive loss of volatile compounds, where recovery of volatile compounds is critical, extract volumes lower than 100 µL are not recommended.

HR Pesticides

Table 6 cont'd...

SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES ALL MATRICES
¹³ C ₆ -Chlorobenzene	10-130
¹³ C ₆ -1,4-dichlorobenzene	15-130
¹³ C ₆ -1,2,3-trichlorobenzene	20-130
¹³ C ₆ -1,2,3,4-tetrachlorobenzene	20-130
¹³ C ₆ -Pentachlorobenzene	20-150
¹³ C ₆ -Hexachlorobenzene	20-150
¹³ C ₆ -beta-HCH	30-150
¹³ C ₆ -delta-HCH ¹	30-150
¹³ C ₆ -gamma-HCH	30-150
¹³ C ₁₀ -Heptachlor	30-150
¹³ C ₁₂ -Aldrin	30-150
¹³ C ₁₀ -Oxychlordane	30-200
¹³ C ₁₀ -trans-Chlordane	30-200
¹³ C ₁₂ -o,p'-DDE	40-150
¹³ C ₁₂ -p,p'-DDE	40-150
¹³ C ₁₀ -trans-Nonachlor	30-150
¹³ C ₁₀ -cis-Nonachlor	30-150
¹³ C ₁₂ -o,p'-DDT	40-150
¹³ C ₁₂ -p,p'-DDT	40-150
¹³ C ₁₀ -Mirex	30-150

QC Parameter	Specification
Analysis Duplicate	Must agree to within $\pm 20\%$ of the mean (applicable to concentrations > 10 times the DL).
Procedural Blank	See Table 6 or $< 10\%$ of analyte value < 0.1 ng for toxaphene.
Instrument Sensitivity	S/N $\geq 3:1$ for 2-4 pg injected for all target analytes (See Table 4). S/N $\geq 2:1$ for 1.6 ng of technical toxaphene.
Instrument Linearity	Linearity is demonstrated by a 5-point calibration over the working concentration range with a relative standard deviation of the RRFs $\leq 20\%$ for targets with a labelled analog present, $\leq 35\%$ for targets with no labelled analog present and all labelled compounds.
Bracketing Cal Ver	RRFs from calibration standards must agree to $\pm 25\%$ over a 12-hour period.
Continuing Cal Ver	Concentrations of native compounds must be within $\pm 20\%$ of expected values for targets with a labelled analog present, $\pm 35\%$ for targets with no labelled analog present. Concentrations of labelled must be within $\pm 35\%$ of expected values.
Chromatogram Quality Max Peak Width: Resolution:	1. Separation between p,p'-DDD and o,p'-DDT $\leq 20\%$ of valley height. 2. p,p'-DDT breakdown must be $\leq 15\%$.
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.
Retention Time Window	RRT must be ± 3 sec of the predicted retention time determined from the calibration standard and adjusted relative to the peak retention time reference (labelled surrogate) Authentic compound must elute after its labelled analogue.
Ion Ratios	Ion ratios must fall within $\pm 20\%$ ($\pm 30\%$ for ¹³ C ₁₀ -Oxychlordane) of the theoretical values for positive identification of all E1 targets in the calibration standards.

¹ delta-HCH normally will elute primarily in the E2 fraction and can be quantified solely from this fraction. Recoveries of ¹³C-delta-HCH may be reported as the sum of the E1 and E2 recoveries if significant concentrations of ¹³C-delta-HCH are observed in the E1 fraction.

HR Pesticides

Table 7. QC Criteria for E2 Chlorinated Pesticides

Analyte:	Typical Sample Specific Detection Limits					Procedural Blank Level ng	Acceptable Matrix Spike % Recovery ¹
	Solid	Aqueous	Tissue	Pulp	XAD column		
	ng/g	ng/L	ng/g	ng/g	ng		
Delta-HCH	0.05	0.5	0.05	0.05	0.5	<0.5	60-130
Heptachlor epoxide	0.05	0.5	0.05	0.05	0.5	<0.5	60-130
alpha-Endosulphan	0.05	0.5	0.05	0.05	0.5	<0.5	70-130
Dieldrin	0.05	0.5	0.05	0.05	0.5	<0.5	60-130
Endrin	0.05	0.5	0.05	0.05	0.5	<0.5	60-130
Endosulphan sulphate	0.05	0.5	0.05	0.05	0.5	<0.5	70-130
Endrin ketone	0.05	0.5	0.05	0.05	0.5	<0.5	60-130
beta-Endosulphan	0.05	0.5	0.05	0.05	0.5	<0.5	70-130
Endrin aldehyde	0.05	0.5	0.05	0.05	0.5	<0.5	50-130
Methoxychlor	0.1	1	0.1	0.1	1.0	<1	60-130
Typical Sample Size:	10 g	1 L	10 g	20 g	1 column		
Typical Final Vol, µL*	200	200	200	200	200		

*Extract volumes may range from 20 µL to 200 µL. To avoid excessive loss of volatile compounds, where recovery of volatile compounds is critical, extract volumes lower than 100 µL are not recommended.

¹Recoveries quoted are guidelines only and vary according to matrix. Consult detailed method performance data available with method documentation for specific criteria.

SURROGATE STANDARD

% RECOVERY RANGES

RECOVERIES:

ALL MATRICES

¹³C₈-delta-HCH
¹³C₁₂-Dieldrin
¹³C₁₂-Endrin
¹³C₁₀-Heptachlor epoxide
¹³C₁₂-Methoxychlor
¹³C₉-alpha-Endosulphan
¹³C₉-beta-Endosulphan

30-150
30-150
30-150
30-150
30-150
30-150
30-150

QC Parameter	Specification
Analysis Duplicate	Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL).
Procedural Blank	See Table 7 or <10% of analyte value.
Instrument Sensitivity	S/N ratio ≤3:1 for 4 pg of all analytes.
Instrument Linearity	Linearity is demonstrated by a 5-point calibration over the working concentration range with a relative standard deviation of the RRFs ≤20% for targets with a labelled analog present, ≤35% for targets with no labelled analog present and all labelled compounds.
Bracketing Cal Ver	RRFs from calibration standards must agree to ±25% over a 12-hour period.
Continuing Cal Ver	Concentrations of native compounds must be within ±20% of expected values for targets with a labelled analog present, ±35% for targets with no labelled analog present. Concentrations of labelled must be within ±35% of expected values.
Chromatogram Quality (GC Resolution)	¹³ C-labelled endrin breakdown must be ≤20%.
Analyte /Surrogate Ratios	Response ratio must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.
Ion Ratios	Ion ratios must fall within ±20% of the theoretical values for positive identification of all E2 targets in the calibration standards (Levels C-F) and samples, and within ±50% for calibration standard Level B. Ion ratio for ¹³ C ₁₂ -Methoxychlor and native methoxychlor are empirically determined from the initial calibration, and ion ratio must be within ±20% for CAL/VER and all samples.

ATTACHMENT 3

STL Richland Gamma Spec SOP

UNCONTROLLED COPY

TITLE: Preparation of All Matrices for Analysis by Gamma Spectroscopy

Prepared by:

Reviewed by:

Approved by:

Approved by:

Approved by:

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Severn Trent Laboratories, Inc.

STL Richland • 2800 George Washington Way, Richland, WA 99352
Tel 509 375 3131 Fax 509 375 5590 • www.stl-inc.com

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Revision 5

R2-0008144

1. SCOPE AND APPLICATION

- 1.1 This procedure describes the preparation of a variety of matrices for analysis by gamma-ray spectroscopy. Depending on the isotopic request, samples are counted either on an Intrinsic (hyperpure) Germanium (HPGE) or Low Energy Photon Detector (LEPD).
- 1.2 The typical isotopes included in the ND library are listed in Appendix B.
- 1.3 This method includes the analysis of the drinking water isotopes, Cs-134, Cs-137 and I-131.
- 1.4 The drinking water detection limits for Cesium-134 and Cesium-137, which are 10 and 20 pCi/L respectively, are met by this procedure.
- 1.5 Refer to Policy P-R-01 for method detection limit information.

2 SUMMARY OF METHOD

- 2.1 A homogenous aliquot of the sample is transferred to a suitable container (matching a standard geometry) and submitted to the counting room for counting on the gamma detectors. HPGE detectors may be used to detect isotopes with gamma-ray energies between 60 and 2000 keV. The LEPD detectors are generally used for isotopes with gamma-ray energies less than 200 keV. Any sample matrix which can be mounted in one of the standard geometries may be analyzed for any of the isotopes included in the Nuclear Data (ND) system software library. Isotopes not included in the ND library may be evaluated manually.
- 2.2 The Low Energy Photon Detector (LEPD) is usually used to identify gamma isotopes with energies less than 200 keV such as I-129, U-238 (Th-234), U-235, Pb-210, Fe-55, Ni-59 and Am-241. Calibrations are developed for specific isotopes as needed. A styrene jar is used for nearly all LEPD counting. Exceptions include liquid geometries greater than 50 mL, some specialty solid geometries, and for some filter (FA) geometries where plastic disks (used to enclose the sample) are wrapped and counted directly on top of the detector.
- 2.3 The volume or weight of sample transferred to the appropriate geometry is recorded. If the sample is to be counted on a LEPD, a weighed aliquot is usually transferred to a styrene jar or the final analysis fraction is sandwiched between a 2" plastic disc and a single layer of plastic wrap for the most efficient geometry. If the sample is to be counted on a HPGE detector, a weighed aliquot is transferred to an appropriately sized container.
 - 2.3.1 Milk: 1000 mL of milk is placed in a 1L Marinelli beaker.
 - 2.3.2 Water: May be counted directly in an appropriate container as received. If a lower detection limit is required, the water is evaporated, transferred into a polyethylene bottle, and counted at a 100 mL (or greater) geometry or transferred to a styrene jar and counted at a 25 or 50 mL geometry.

2.3.3 Urine: urine and other liquids are transferred to an appropriate sized container and diluted, if necessary, to a standard volume. (These may also be concentrated to meet a lower detection limit, as for water, if requested).

2.3.4 All other samples for gamma spectroscopy analysis are transferred to an appropriately sized container and the nearest geometry is estimated and noted on the analytical work sheet.

3 DEFINITIONS

3.1 HPGE – Intrinsic (hyperpure) Germanium Detector

3.2 LEPD – Low Energy Photon Detector

4 INTERFERENCES

4.1 Whenever an isotope is quantified by the gamma emission of one of its progeny, the assumption of equilibrium between the parent and its progeny is necessary. The sample usually must be allowed some time after being transferred to the counting container to reattain equilibrium before counting. Ra-226, U-234, and U-238 are three isotopes for which it is necessary to specify ingrowth holding times after the sample is placed in the counting container. Ra-226 may be determined without an ingrowth holding time if the 186 keV line is used. The 186 keV line for Ra-226 may only be used with a limited number of geometries, and only if the proper library is specified. Caution must be exercised in using the 186 keV line to determine Ra-226, as U-235 may interfere. Associates should consult supervision if they are uncertain of how to proceed.

5 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1 Specific Safety Concerns or Requirements

- All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported immediately to a laboratory supervisor.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Octanol	Flammable		Liquid may be irritating to skin and eyes. Store in a cool, dry, well-ventilated, flammable liquid storage area or cabinet. Vapors may flow along surfaces to distant ignition sources and flash back.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6 EQUIPMENT AND SUPPLIES

- 6.1 Aluminum cans - 95 mL capacity with lids and can sealer.
- 6.2 Bags - plastic.
- 6.3 Balance - top loading, able to read to nearest whole gram.
- 6.4 Beakers - glass, large enough to contain a water sample.
- 6.5 Bottles - polyethylene, wide mouth, 450 mL capacity and polyvinyl chloride, narrow mouth, 500 mL capacity, with screw caps. The 450 mL bottle is obtained from Quintex Corporation or equivalent.
- 6.6 Flasks - Erlenmeyer, various sizes.
- 6.7 Funnels.
- 6.8 Hot plate.
- 6.9 Marinelli beakers - 450 (MA) and 1000 (MB) mL capacity.

- 6.10 Petri dish - 60 mm, plastic.
- 6.11 pH paper - 0 to 14 range.
- 6.12 Plastic wrap.
- 6.13 Styrene jars - plastic 250 mL capacity, 2½" diameter by 3½" in height, wide mouth with lids.
- 6.14 Vinyl tape - various colors.

7 REAGENTS AND STANDARDS

- 7.1 Reagents are prepared from analytical reagent grade chemicals unless otherwise specified below. Reagent water, which must have an electrical resistivity of 1 megohm-cm or greater when obtained, is used throughout. Reagent water is obtained from the Nanopure system. Label all reagents as outlined in procedure RICHQA5002.

NOTE: Consult the Material Safety Data Sheets for the properties of these reagents and how to work with them.

- 7.2 14.7M Ammonium hydroxide (14.7M NH_4OH) - Concentrated. **CAUTION:** Corrosive.
- 7.3 30% Hydrogen peroxide (30% H_2O_2). **WARNING:** Corrosive and Oxidizer.
- 7.4 16M Nitric acid (16M HNO_3) - Concentrated. **WARNING:** Corrosive and Oxidizer.
- 7.5 8M Nitric acid (8M HNO_3) - Add 500 mL 16M HNO_3 to approximately 400 mL reagent water. Dilute to 1L with reagent water and mix well. **WARNING:** Corrosive and Oxidizer.
- 7.6 10% Sodium bisulfite (10% NaHSO_3) - Dissolve 1.0g NaHSO_3 in 9 mL of reagent water. Prepared fresh for each use.
- 7.7 12M Sodium hydroxide (12M NaOH) Slowly dissolve 480 g of sodium hydroxide pellets in approximately 400 mL of reagent water. Dilute to 1 L with reagent water and mix well. Or, use the commercially prepared 50% NaOH solution. **WARNING:** Corrosive. Do not store for a long time due to CO_2 absorption which forms carbonate (CO_3^{2-}).
- 7.8 Phenolphthalein Indicator 0.1% - Dissolve 100 mg phenolphthalein in 60 mL of ethyl alcohol. Dilute to 100 mL with reagent water. Mix well. **WARNING:** Flammable.

7.9 Octanol - **WARNING:** Flammable.

8 SAMPLE COLLECTION, PRESERVATIVES, SHIPMENT AND STORAGE

- 8.1 The sample may be collected in glass or plastic containers using no preservatives. Storage of the sample prior to analysis should not exceed six months.
- 8.2 It is recommended that water samples be preserved at the time of collection by adding enough 1N HNO₃ to the sample to bring it to pH 2.

9 QUALITY CONTROL

- 9.1 All quality control data shall be maintained and available for easy reference.
- 9.2 Consult the Quality Assurance Summary for client specific information regarding QC frequency.
- 9.3 An appropriate blank shall be prepared in the same geometry as that of the batch of samples to be counted. The sample date on the analytical work sheet shall be the date of preparation of the blank sample.
- 9.4 An appropriate spike shall be prepared in the same geometry as that of the batch of samples to be counted. Consult the latest version of the client specific Quality Assurance Summary (QAS) for the appropriate spike activity to use. The spike may be prepared in one of two ways.
 - 9.4.1 The spike may be prepared by the addition of the contents of a spike vial to an aliquot of reagent water, which is then diluted to the appropriate geometry volume. For a spike prepared in this manner, the sample date on the analytical work sheet shall be the preparation date of the spike vial except for Sr-85 vials where the reference date is used. The proper vial code to be used in preparation of a spike will depend upon contract criteria.
 - 9.4.2 A prepared spike may be reusable (as in the soil laboratory control sample containing Cs-134, Cs-137, Ra-226 and Ra-228). For this type of spike, the sample date on the analytical work sheet shall be the first day of the quarter of the year in which the sample being counted (ie., 1JanXX, 1AprXX, 1JulXX, 1OctXX).
- 9.5 Refer to SOP RICH-RC-0002 for sample and QC data acceptance criteria and corrective action.

10 CALIBRATION AND STANDARDIZATION

- 10.1 Germanium detectors are calibrated according to RICHQA5015, Efficiency Calibration of Germanium Spectroscopy Detectors.

11 PROCEDURE

NOTE: If any parameter is found to be out of limits, consult supervision. Also, a nonconformance will be issued to the Quality Assurance Group.

NOTE: One time procedural variations are allowed if deemed necessary by the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size or other parameters. Any variation in procedure shall require approval by supervision and immediate notification of the Quality Assurance Group. If contractually required, the client shall be notified prior to any procedure changes. A Nonconformance Memo shall be completed and forwarded to the Quality Assurance Group within one day of the supervisor's approval. The Nonconformance Memo will be filed in the project file.

- 11.1 **CAUTION:** When a sample has been placed in a container and sealed and labeled, the container should be kept in a upright position at all times. Tipping the sample container onto its side may cause some sample to be trapped up by the lid making the geometry different from the standard geometries for which calibrations have been performed.

Tables 1 and 2 in Appendix A, show the appropriate container, and volume/weight of sample for existing geometries for the HPGE and LEPD detectors.

11.1.1 Milk

- 11.1.1.1 Shake the sample container(s). Transfer 1000 mL of milk to a 1 L Marinelli beaker. Tap the lid to remove the air from above the sample and tape the lid securely with vinyl tape. Place the sample in the refrigerator. Record the sample volume and geometry on the analytical work sheet. Proceed to step 11.2.

11.1.2 Water, Urine, and other liquids

11.1.2.1 For Direct Counting:

- 11.1.2.1.1 The sample may be counted directly by transferring a sample aliquot to a counting container to match the largest standard geometry possible as directed by the client specific QAS and recording the aliquot weight/volume and geometry on the analytical work sheet. Proceed to 11.2.

NOTE: The detection limit can be significantly improved by concentrating the sample through evaporation.

11.1.2.2 For concentration of water and aqueous liquids:

- 11.1.2.2.1 Before concentrating, the pH must be less than 2. Verify that the pH is 2 or less in the original sample container. If the pH is greater than 2, acidify the

samples using 16M HNO₃ until the sample pH is less than 2. If acidification was necessary, allow the sample to sit for at least sixteen hours in the original container before proceeding.

- 11.1.2.2.2 Mix the sample prior to measuring out an aliquot (specified in the client specific QAS) to an appropriately sized and labeled beaker. Evaporate the sample on a hot plate to a volume appropriate for the final geometry to be used. Consult the client specific QAS for geometry specifications. If the geometry is not specified in the QAS, consult supervision. Allow the sample to cool to room temperature. Proceed to 11.1.2.6.

11.1.2.3 For concentration of water and aqueous liquids for I-131:

- 11.1.2.3.1 Before concentrating, the pH must be less than 2. Verify that the pH is 2 or less in the original sample container. If the pH is greater than 2, acidify the samples using 16M HNO₃ until the sample pH is less than 2. If acidification was necessary, allow the sample to sit for at least sixteen hours in the original container before proceeding.
- 11.1.2.3.2 Mix the sample prior to measuring out an aliquot (specified in the client specific QAS) to an appropriately sized and labeled beaker. Add the appropriate spike and/or yield monitors to each beaker. Rinse the yield monitor vials 3 times with reagent water and the spike vials with 2M HNO₃. Remove the labels from the QC vials and place them on the analytical work sheet.
- 11.1.2.3.3 Add approximately 2 mL of freshly prepared 10% NaHSO₃ and approximately 3-4 mL of 12M NaOH to all the beakers except the LCS. Add a few drops of phenolphthalein indicator. The solution should be pink, pH>9. If not, add a few more drops of NaOH. Evaporate the sample on a hot plate to a volume appropriate for the final geometry to be used. Consult the client specific QAS for geometry specifications. If the geometry is not specified in the QAS, consult supervision. Allow the sample to cool to room temperature. Proceed to 11.1.2.6.

11.1.2.4 For concentration of Urine:

- 11.1.2.4.1 Pour the sample into a labeled flask/beaker and rinse the plastic collection bottle with concentrated HNO₃, unless already acidified. Add the rinses to the flask/beaker using a total volume of acid approximately equivalent to 10% of the sample volume.

11.1.2.4.2 Measure out an aliquot (specified in the client specific QAS) to an appropriately sized and labeled beaker. Add about 1%, by volume of 30% H_2O_2 and 1-2 drops of Octanol. Digest and evaporate the sample on a hot plate to a volume appropriate for the final geometry to be used. Consult the client specific QAS for geometry specifications. If the geometry is not specified in the QAS, consult supervision. Allow the sample to cool to room temperature. Urine samples may be taken just to dryness over gentle heat and the residue redissolved with 8M HNO_3 . Proceed to 11.1.2.6.

11.1.2.5 **NOTE:** If Ra-226 is requested and the 186 keV Ra-226 gamma line is not used, the sealed Aluminum can geometry must be used. Samples must be held for seventeen days after sealing, for ingrowth, before counting.

If the sealed Aluminum can geometry is to be used, continue with this step, otherwise go to 11.1.2.6. The sample should be evaporated to approximately 50 mL. The pH of the sample shall be adjusted to a pH of 7 ± 0.5 with concentrated NH_4OH . Pour the sample into a labeled can and rinse the evaporation container with 2-3 small rinses of reagent water (total volume < 95 mL). Add reagent water till the total volume is 95 mL and seal the can. Proceed to step 11.2.

CAUTION: Do not overfill can or leakage during sealing will occur.

11.1.2.6 Pour the sample into the appropriate container. Rinse the evaporation container with 2-3 small rinses of reagent water and pour the rinsate into the counting container. Add reagent water to bring the total volume to the appropriate geometry. This volume is measured by comparing the sample container to a pre-marked container. Cap the container and seal the cap with vinyl tape. Clearly mark the sample container with the STL Richland sample number and place the container in a plastic bag (or double bag if so directed). Proceed to step 11.2.

11.1.3 Feces

11.1.3.1 Fecal samples may be counted as received, or may be digested prior to counting.

11.1.3.2 For counting as received, place a 450-mL wide mouth polyethylene bottle on the balance and tare the balance. Transfer the total fecal sample to the bottle and record the weight on the analytical sheet. Sample sizes less than 100 mL geometry equivalent should be placed in a styrene jar. If the sample is received in a plastic bag, place the sample, still in the bag, in the counting container. Compress the sample into the corners of the bottle and level by compressing or gently tapping. Seal the container with vinyl tape. Clearly mark the sample container with the STL Richland sample number. Estimate to the nearest standard geometry and record on the analytical work sheet. Proceed to step 11.2.

- 11.1.3.3 For counting after digestion, place a two-liter beaker on the balance and tare the balance. Transfer the sample to the beaker and record the sample weight on the analytical worksheet. Add sufficient octanol to just coat the surface of the sample. Add 200-300 mL of 8M HNO₃ and 40-50 mL of 30% H₂O₂ to the beaker. Heat over gentle heat until the sample just goes to dryness. Wet ash the sample with concentrated HNO₃ and 30% H₂O₂ until the sample is a whitish purple color. Add approximately 80 mL of 8M HNO₃ and warm gently. Transfer the sample to a counting container using reagent water and dilute to the desired standard geometry. This volume is measured by comparing the sample container to a pre-marked container. Cap the container and seal the cap with vinyl. Clearly mark the sample container with the STL Richland sample number and place the container in a plastic bag (or double bag if so directed). Record the geometry on the analytical work sheet and proceed to step 11.2.

11.1.4 Soil and vegetation

- 11.1.4.1 **NOTE:** If U-234 determination is requested by gamma spectrometry, consult supervision for the appropriate geometry.

NOTE: If Ra-226 if requested, hold the sample for 10 days after placing in the counting container but prior to counting for ingrowth.

Dry and sieve the sample as in RICHRC5016 or RICHRC5013, if requested. Place an empty container of the appropriate size as stated in the client specific QAS, usually a plastic Marinelli beaker (450-mL or 1000-mL, depending on sample size), on the balance and tare the balance. Transfer an aliquot large enough to fill the container to within 3/4" of the top. Pack vegetation samples but allow to re-expand when adjusting to 3/4" of the top and record the weight of sample on the analytical worksheet. Replace the lid and seal with vinyl tape. Write the STL Richland sample number on a piece of white vinyl tape and place it on the top of the lid. Record the appropriate geometry on the analytical work sheet and proceed to 11.2.

- 11.1.4.1.1 If the sample volume is less than that required to fill the appropriate client specific geometry, then use a wide mouth plastic bottle or styrene jar and fill to the largest geometry possible and record the sample weight on the analytical worksheet. Seal with vinyl tape and record the geometry on the analytical work sheet. Clearly mark the STL Richland sample number on the bottle or jar and place in a plastic bag. Proceed to step 11.2.
- 11.1.4.1.2 Our experience has shown that for environmental levels of Ra-226 in soil, a 10 day ingrowth period after drying and grinding will restore equilibrium. Therefore, unless a sealed container is specified by contract or Ra-226 levels in excess of 10X environmental levels are expected, the sealed aluminum can geometry is not required.

If the sealed Aluminum can geometry is to be used, continue with this step. Weigh the empty can. Fill the can with sample and reweigh the filled can. Record the sample weight on the analytical worksheet. Seal the lid on the can and label the can. Proceed to step 11.2.

CAUTION: Do not overfill can or leakage during sealing will occur.

11.1.5 Air Filters (47mm)

11.1.5.1 For single filters: Label a 60 mm plastic petri dish and place the filter inside. Replace the lid and tape around the edge of the dish with vinyl tape. Clearly mark the sample container with the STL Richland sample number. Record the appropriate sample size analyzed (cubic meters, 1 sample, etc) and geometry on the analytical work sheet. Proceed the step 11.2.

11.1.5.2 For multiple filters: Label a styrene jar and place filters in the jar. Estimate the nearest geometry, 25 mL, 50 mL, depending on the number of filters analyzed. Seal the container with vinyl tape. Clearly mark the sample container with the STL Richland sample number. Record the appropriate sample size analyzed (cubic meters, 1 sample, etc) and geometry on the analytical work sheet. Proceed to step 11.2.

11.1.5.2.1 For certain clients, filters may be placed in a styrene jar with sufficient 2M HNO₃ to fill a specific geometry's volume. Allow several hours for leaching and swirl to distribute any leached material throughout the liquid prior to counting.

11.1.5.2.2 For certain clients, filters are digested and the resulting solution filtered. The filtrate is evaporated to a desired geometry and transferred to an appropriate counting container.

11.1.6 Charcoal

11.1.6.1 Place a charcoal filter, removed from its plastic bag, in a labeled, styrene jar. Make sure the filter flow arrow is pointing upward. Record the sample size in cubic meters and specify "charcoal filter" on the analytical work sheet. Charcoal filter samples are marked with the STL Richland sample number. Place the styrene jar in a plastic bag. Proceed to step 11.2.

11.1.7 Meat and Produce

11.1.7.1 Small sample sizes may be placed directly in a pre-weighed styrene jar and the appropriate geometry selected. Larger samples of meat and produce are cut into small sections and packed in a pre-weighed, Marinelli beaker to within ¾" of the top, or in a pre-weighed wide mouth plastic bottle or styrene jar to the largest geometry

possible. Weigh the container with the sample and record the net weight of sample and the geometry on the analytical work sheet. Seal the sample container with vinyl tape. Clearly mark the STL Richland sample number on the sample container. Place styrene jars or bottles in plastic bags. Place samples in the refrigerator. Proceed to step 11.2.

NOTE: For egg samples, remove the shells and discard them (unless Sr or another analysis is requested on the shells) before placing sample in the container.

11.1.7.2 Produce - I-129

11.1.7.2.1 Wet and dry weights of the entire sample must be noted and recorded, as applicable, as described in RICHRC5016. A styrene jar is placed on the balance and the balance is tared. An appropriate aliquot of the sample is transferred to the styrene jar and compressed lightly. IV9 geometry is noted on the analytical work sheet.

11.1.7.2.2 Clearly mark the sample container with the STL Richland number of the sample. Seal with vinyl tape. Place the filled counting container in a plastic bag. Proceed to step 11.2.

11.1.8 Resin and Filters

11.1.8.1 The preparation of resin samples and filters are covered in RICHRC5045, Preparation of Resin and Filter Samples.

11.2 Verify that the analytical work sheets contain all necessary information (sample analyzed, sample date, geometry, detector type, and counting time). The completed analytical work sheets and samples (if not refrigerated) are submitted to the counting room. Refrigerated samples are removed from the refrigerator by the counting room personnel as needed and returned to the refrigerator after counting.

NOTE: If ingrowth is required, note the desired count date on the analytical work sheet before submitting to the counting room. Alert the counting room staff that ingrowth is required.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Gamma activity calculations are performed by computer using Nuclear Data (ND) and/or internally developed software. Further information is provided in ND manuals software documentation files.

12.2 Wet Weight Conversion Equation:

$$\frac{D (g)}{1 - M} = W (g)$$

where:

D = dry weight of the sample aliquot.

M = percent moisture expressed as a decimal.

W = converted wet weight of the sample aliquot.

13 METHOD PERFORMANCE

13.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use.

13.2 Method Demonstration of Capability documentation is maintained in the quality files.

14 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Aqueous acidic waste pH < 2. Waste is collected in an Acid Waste container color coded with Yellow tape and a Yellow cap.
- Aqueous Basic waste with a pH > 2. Waste is collected in a Neutral waste container color coded with Green tape and a Green Cap
- Flammable waste. Waste is collected in a Flammable waste container which is red with a self closing metal lid

Radioactive Waste

Dry Radioactive waste shall be placed in a RAD Trash container. This waste stream can be distinguished by its yellow trash bags, and radioactive material stickers on the trash container.

All mixed waste shall be placed in the appropriate radioactive waste stream. The RAD waste stream color codes are the same as the non-radioactive waste streams but there are radioactive material stickers on the SAP container and spill tray.

Broken glassware shall be deposited in the appropriate broken glassware container. All Category I broken glassware is deposited in a White Broken Glass container. All Category II and/or III broken glassware is deposited in Magenta with White Radioactive Material box.

15 REFERENCES

- 15.1 National Council on Radiation Protection and Measurements. A Handbook of Radioactivity Measurement and Procedures. NCRP Report 58 and Edition, Washington, D.C., 1985.
- 15.2 American Society for Testing and Materials. Standard Practice for High-Resolution Gamma-ray Spectrometry of Water in Annual Book of ASTM standards. Philadelphia, PA: ASTM D3649-91, 1992.
- 15.3 Chieco, N.A., D.C. Bogen, and E.O. Knutson. eds. 1990 EML Procedures Manual, Gamma, HASL-300, 27th Edition, Volume1. Environmental Measurements Laboratory, US Department of Energy, New York, New York.
- 15.4 U.S. Environmental Protection Agency, August 1980, "Gamma Emitting Radionuclides in Drinking Water", Method 901.1, EPA-600/40-80-032, National Technical Information Services, Springfield, VA.
- 15.5 STL Quality Management Plan, latest revision.
- 15.6 STL Richland Laboratory Quality Manual, latest revision.
- 15.7 STL Richland Quality Assurance Summary, latest revision.
- 15.8 Associated SOPs
 - 15.8.1 RICHQA5002 - Chemical/Reagent Labeling.
 - 15.8.2 RICHQA5015 - Efficiency Calibration of Germanium Spectroscopy Detectors.
 - 15.8.3 RICHRC5013 - Preparation of Soil Samples.
 - 15.8.4 RICHRC5016 - Preparation of Environmental Matrices.
 - 15.8.5 RICHRC5045 - Preparation of Resin and Filter Samples.

16 MISCELLANEOUS

16.1 Responsibilities

Analyst: Implements SOP as written.

Counting Room: Performs review on raw instrument data.

Technical Data Reviewer: Performs final data review.

Project Manager: Confirms final review and prepares data for reporting to client.

QA Manager: Performs product quality assessments as defined in the Quality Assurance procedures.

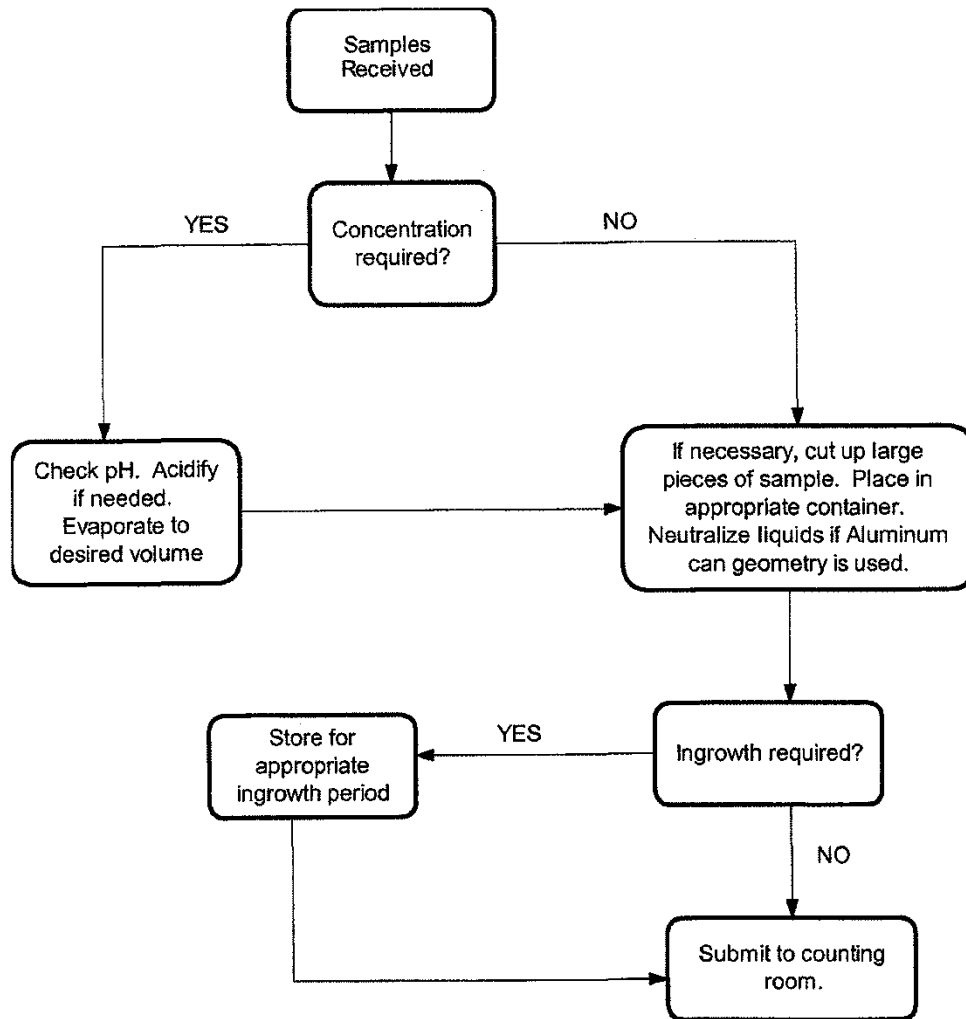
16.2 Records Management/Documentation

16.2.1 All records generated by this analysis will be filed and kept in accordance with STL Richland SOPs for records management and maintenance.

16.3 Drinking Water Method Variations

17.3.1 This procedure is compliant with Method 901.1. RICH-RC-5017 includes the use of HPGEs and LEPDs.

16.4 Procedural Flow Chart



APPENDIX A

Table 1. Geometries for the HPGE Detectors

Geometry	Description
FP	one filter in the bottom of a 60 mm Petri dish.
25 mL	25 mL of liquid in a styrene jar.
25 mL_SE75	Se-75 in 25 mL of liquid in a styrene jar.
50 mL	50 mL of liquid in a styrene jar
100 mL	100 mL of liquid in a 500 mL polybottle.
200 mL	200 mL of liquid in a 500 mL polybottle.
500 mL	500 mL of liquid in a 500 mL polybottle.
CHR	I-131 in a charcoal filter placed in a styrene jar.
MA	450 mL Marinelli beaker.
MB	1 L Marinelli beaker.
ROFS	200 mL of soil in a wide mouth plastic bottle.
25ROF	25 mL of soil in a styrene jar.
S25	25 mL of soil in a styrene jar.
S200	200 mL of soil in a 500 mL polybottle.
SMA	Soil in a 450 mL Marinelli beaker.
WALCAN	Sealed aluminum can for liquids.
ALCAN	Sealed aluminum can for soils.

NOTE: The 450 mL wide-mouth plastic bottle obtained from Quintex Corporation (P9-35) is considered equivalent to the 500 mL polybottle. The wide mouthed plastic bottle is used for counting dry matrices using the 100 mL through 500 mL HPGE geometries.

500 mL polybottle - polyvinyl chloride of 500 mL capacity, boston round shape, 78 mm outside diameter and 115 mm in height to the shoulder of the bottle.

Styrene jar - 250 mL capacity plastic jar, 60-63 mm inside diameter, 80-82 mm inside depth.

Table 2 - Geometries for the LEPD Detectors

Geometry	Description
FA	filter/plastic disc sandwich.
25 mL	25 mL of liquid in a styrene jar.
50 mL	50 mL of liquid in a styrene jar.
100 mL	100 mL of liquid in a 500 mL polybottle.
200 mL	200 mL of liquid in a 500 mL polybottle.
400 mL	400 mL of liquid in a 500 mL polybottle.
500 mL	500 mL of liquid in a 500 mL polybottle.
IC9 or IC5	I-129 and I-125 - charcoal filter in a styrene jar.
I_FA	I-129 in a filter/plastic disc sandwich.
IS9	I-129 in 50 grams of soil in a styrene jar.
IV9	I-129 in 50 grams of alfalfa in a styrene jar.
IW9	I-129 in 25 mL of water in a styrene jar.
PBA	Pb-210 in 25 grams of soil in a styrene jar.
PBC	Pb-210 in 90 gram geometry.
UGS	U-Nat in 100 grams of soil in a styrene jar.
X_RAY	Fe-55 and Ni-59 in a filter/plastic disc sandwich.

NOTE: The 450 mL wide-mouth plastic bottle, obtained from Quintex Corporation (P9-35), is considered equivalent to the 500 mL polybottle. The wide mouthed plastic bottle is used for counting dry matrices using the 100 mL through 500 mL HPGE geometries.

500 mL polybottle - polyvinyl chloride of 500 mL capacity, boston round shape, 78 mm outside diameter and 115 mm in height to the shoulder of the bottle.

Styrene jar - 250 mL capacity plastic jar, 60-63 mm inside diameter, 80-82 mm inside depth.

Filter/plastic sandwich - filter on a plastic disk and wrapped tightly in a single layer of plastic wrap.

Appendix B
Gamma Isotopes from ND Library
(EXAMPLE)

ISOTOPE	HALF-LIFE	KEY LINE ENERGY
Be-7	53.44 days	477.59 keV
Na-22	2.60 years	1274.54 keV
Na-24	15.00 hours	1368.53 keV
K-40	1.28E+09 years	1460.81 keV
Sc-46	83.85 days	1120.51 keV
Cr-51	27.70 days	320.08 keV
Mn-54	312.70 days	834.83 keV
Co-57	270.90 days	136.48 keV
Co-58	70.80 days	810.76 keV
Fe-59	44.63 days	1099.22 keV
Co-60	5.27 years	1332.49 keV
Zn-65	244.40 days	1115.52 keV
Se-75	119.78 days	264.65 keV
Sr-85	64.84 days	513.99 keV
Y-88	106.61 days	1836.01 keV
Nb-94	20300.00 years	871.10 keV
Nb-95	35.06 days	765.79 keV
Tc-95M	61.00 days	204.12 keV
Zr-95	64.02 days	756.72 keV
ZrNb-95	64.40 days	765.82 keV
Mo-99	66.02 hours	140.50 keV
Ru-103	39.35 days	497.08 keV
Ru-106DA	368.20 days	621.84 keV
Ag-108M	127.00 years	433.93 keV
Ag-110M	249.85 days	884.67 keV
Sn-113DA	115.10 days	391.69 keV
Sb-124	60.20 days	602.72 keV
Sb-125	2.77 years	427.89 keV
Sn-126DA	1.00E+05 years	666.33 keV
I-131	8.04 days	364.48 keV
Ba-133	10.50 years	356.00 keV
Cs-134	2.06 years	604.70 keV
Cs-137DA	30.17 years	661.65 keV
La-138	1.06E+11 years	1435.80 keV
Ce-139	137.20 days	165.85 keV
Ba-140	12.79 days	537.32 keV
BaLa-140	12.79 days	1596.20 keV

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La-140	40.22 hours	1596.49 keV
ISOTOPE	HALF-LIFE	KEY LINE ENERGY
Ce-141	32.50 days	145.44 keV
Ce-144	284.30 days	133.54 keV
CePr-144	284.20 days	133.53 keV
Pm-144	363.00 days	618.06 keV
Pm-146	5.53 years	453.89 keV
Eu-152	13.60 years	344.27 keV
Eu-154	8.80 years	1274.45 keV
Eu-155	4.96 years	105.31 keV
Hf-181	42.39 days	482.03 keV
Bi-207	33.40 years	569.67 keV
Tl-208	1.41E+10 years	583.14 keV
Bi-210M	3.00E+06 years	266.15 keV
Pb-212	1.41E+10 years	238.63 keV
Bi-214	4.47E+09 years	609.31 keV
Pb-214	4.47E+09 years	351.92 keV
Ra-223	21.77 years	269.46 keV
Ra-224DA	1.91 years	238.63 keV
Ra-226DA	1600.00 years	609.32 keV
Ac-227DA	21.77 years	236.00 keV
Ac-228	1.41E+10 years	911.07 keV
Ra-228DA	5.75 years	911.07 keV
Th-228DA	1.91 years	583.14 keV
Th-232DA	1.41E+10 years	338.32 keV
Th-234DA	4.47E+09 years	1001.03 keV
U-234DA	4.47E+09 years	295.22 keV
U-235HP	7.04E+08 years	143.76 keV
Np-237DA	2.14E+06 years	311.98 keV
U-238DA	4.47E+09 years	351.92 keV
U-238DHP	4.47E+09 years	63.28 keV
Am-241HP	432.20 years	59.54 keV

R2-0008163

ATTACHMENT 4

STL Burlington Mercury SOP

1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes the laboratory procedure for the analysis of total mercury in water, sediment, sludge and soil samples taken from hazardous waste sites using a cold vapor technique with atomic absorption (AA).
- 1.2 The normal range of determination may vary by instrument conditions and/or increasing/decreasing sample size. For aqueous samples, the contract required detection limit (CRQL) is 0.2ug/L, and for soils, the CRQL is 0.1mg/kg.

2.0 SUMMARY OF METHOD**2.1 Water Samples**

A measured volume of sample is acid digested with potassium permanganate and potassium persulfate for two hours in a block digester maintained at a temperature of 95°C. After digestion, hydroxylamine hydrochloride is added to each digestate in order to reduce excess permanganate. The digestate is placed on a closed-system mercury autoanalyzer and stannous chloride is added to each sample. The elemental mercury released is measured spectrophotometrically at a wavelength of 253.7 nm. The concentration is calculated from the response of the sample absorbance applied against the calibration curve.

2.2 Soil/Sediment Samples

A weighed portion of solid sample is acid digested for 2 minutes at a temperature of 95°C then digested with potassium permanganate and potassium persulfate for 30 minutes at a temperature of 95°C. Hydroxylamine hydrochloride is added to each digestate in order to reduce excess permanganate. The digestate is placed on a closed-system mercury autoanalyzer and stannous chloride is added to each sample. The elemental mercury released is measured spectrophotometrically at a wavelength of 253.7 nm. The concentration is calculated from the response of the sample absorbance applied against the calibration curve.

- 2.3 This procedure is based on CLP SOW ILM05.3, Exhibit D (Mercury).

3.0 DEFINITIONS

- 3.1 A list of definitions is given in Appendix B.

4.0 INTERFERENCES**4.1 Water**

Some seawaters and wastewaters that are high in chlorides exhibit a positive interference. During the oxidation step chlorides are converted to free chlorine which will also absorb radiation at 253 nanometers (nm). To ensure free chlorine is absent before the mercury is

reduced and swept into the cell use an excess of hydroxylamine HCl reagent (25mL) and additional amounts of permanganate [as much as 25 milliliters (mL)].

The formation of a heavy precipitate, in some wastewaters and effluents, has been reported upon addition of concentrated sulfuric acid. If this is encountered, the sample cannot be analyzed by this procedure.

Possible interference from sulfide is eliminated by the addition of potassium permanganate. Copper has also been reported to interfere; however, per the CLP SOW, copper concentrations as high as 10mg/L have not shown an effect on recovery of mercury from spiked samples.

If total mercury values are to be reported, water samples that contain solids must be blended and then mixed during field sampling.

4.2 Soil/Sediment

The same types of interferences noted for water samples are possible with sediments.

Samples that contain high concentrations of oxidizable organic materials, (high COD values) may not be completely oxidized by this procedure. If this occurs, the recovery of organic mercury will be low. To eliminate the problem, reduce the weight of the original samples or increase the amount of potassium persulfate (and stannous chloride) used in the digestion.

5.0 SAFETY

- 5.1. Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added. Protective clothing such as a lab coat, safety glasses and latex gloves must be worn while performing this procedure.

5.3. Primary Materials Used

Table 1, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.



6.0 EQUIPMENT AND SUPPLIES

- 6.1 Mercury Auto-Analyzer; Leeman Labs PS 200; Leeman Labs PS 200II, Leeman Labs Hydra AA.
- 6.2 Block digester; Environmental Express
- 6.3 Polyethylene Digestion Vessels; 100 mL; Environmental Express
- 6.4 Volumetric Pipets
- 6.5 Top Loading Balance

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent Water, ASTM Type II

Sulfuric Acid, concentrated - Reagent grade; JT Baker or equivalent

Nitric Acid, concentrated - Reagent grade; JT Baker or equivalent

Stannous Chloride; Reagent grade; JT Baker or equivalent

Hydroxylamine Hydrochloride; Reagent grade; JT Baker or equivalent

Potassium Permanganate; Reagent grade; JT Baker or equivalent

Potassium Persulfate; Reagent grade; JT Baker or equivalent

Stannous Chloride: Add 100g of stannous chloride to 1L of 10% hydrochloric acid.

Hydroxylamine Hydrochloride: Dissolve 240grams of Hydroxylamine Hydrochloride in 2L of reagent water.

Potassium Permanganate (KMNO_4): 5% solution w/v: Dissolve 100 grams of Potassium Permanganate in 2L of reagent water.

Potassium Persulfate – 5% solution w/v: Dissolve 100 grams of Potassium Persulfate in 2L of reagent water.

7.2 Standards

Stock standard solutions are purchased from commercial vendors. Intermediate and working standard solutions are prepared from stock standards by diluting volumes of stock standards with acidified reagent water in volumetric flasks to obtain the appropriate concentration. Expiration dates of prepared solutions are assigned according to expiration

date of the parent standard. Standard solutions are prepared as needed or on the expiration date, whichever occurs first.

Mercury Stock Standard Solution (1000mg/L): Purchased from Spex.

Mercury Intermediate Standard (10,000ug/L): Add 1mL of the 1000mg/L stock standard solution and 0.15mL of concentrated HNO₃ to a 100mL volumetric flask that contains approximately 80mL reagent water. Adjust to volume with reagent water.

Mercury Working Standard (100ug/L): Add 10mL of the intermediate standard and 1.5mL of concentrated HNO₃ to a 1000mL volumetric flask that contains approximately 800mL reagent water. Adjust to volume with reagent water. Secondary dilutions of the working standard are used for the calibration curve, continuing calibration verification (CCV) standards, and the Contract Required Quantitation Limit (CRQL) Check Standard (CRI).

ICV Stock Standard Solution (1000mg/L): Purchased from Inorganic Ventures.

ICV Intermediate Standard Solution (10,000ug/L): Add 1mL of the 1000mg/L ICV stock standard solution and 0.15mL of concentrated HNO₃ to a 100mL volumetric flask that contains approximately 80mL reagent water. Adjust to volume with reagent water.

ICV Working Standard Solution (30ppb): Add 1.5mL of the ICV intermediate standard and 0.75mL of concentrated HNO₃ into a 500mL volumetric flask that contains approximately 300mL reagent water. Adjust to volume with reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

- 8.1 All samples should be collected in glass or polyethylene containers and iced or refrigerated to 4°C (±2°C) from the time of collection until digestion. Water samples must be preserved nitric acid to pH less than 2 immediately after collection. If dissolved metals are to be determined, the samples must be filtered through a 0.45 micrometer (µm) pore diameter membrane filter at the time of collection or as soon as possible. Preserve the filtrate with nitric acid to pH less than 2 immediately after filtration.
- 8.2 The maximum holding time for mercury is 26 days from Validated Time of Sample Receipt (VTSR).
- 8.4 Samples are stored protected from light and refrigerated at 4°C (± 2°C) from the time of receipt until 60 days after delivery of a reconciled, complete sample data package to the client. After 60 days, the samples are disposed of in a manner that complies with all applicable regulations.

9.0 QUALITY CONTROL

9.1 Preparation Blank (PB)

A PB is prepared and analyzed with each digestion batch or every sample delivery group, whichever is more frequent. The concentration of mercury in the blank must be less or

equal to the CRQL. If this criterion is not met, the lowest concentration of mercury in the samples must be greater than or equal to 10 times the concentration in the blank. Otherwise, the blank and associated samples are re-digested and re-analyzed. If the concentration of the blank is below the negative CRQL, all samples associated with the blank that have mercury concentrations below 10 times the CRQL are re-digested and re-analyzed.

9.2 Laboratory Control Sample (LCS)

A solid laboratory control sample is prepared and analyzed with each soil digestion batch. The percent recovery of the LCS must be within the control limits specified for the standard reference material (usually 85-115%). If the results are not within the control limits, the problem should be investigated, corrected, and the associated samples must be re-digested and re-analyzed along with the LCS and preparation blank.

9.3 Matrix Spike (MS)

At least one matrix spike is prepared and analyzed with each digestion batch or every sample delivery group whichever is more frequent. The MS provides information about the effect of the sample matrix on the digestion or analysis process and is analyzed in the same manner as the unspiked sample. The percent recovery of the MS should be within the limits of 75-125%. If it is not, the results of associated sample data are flagged with an "N" on the deliverable forms unless the sample concentration exceeds the value of spike added by a factor of four or more. In the latter event, the data is reported without qualification.

9.4 Sample Duplicate (SD)

One duplicate sample is prepared and analyzed from each group of samples of a similar matrix. The relative percent difference between duplicate sample results greater than or equal to the CRQL should be less than or equal to 20%. If the results are less than 5 times the CRQL, a control limit of the CRQL is reported on the deliverable forms, otherwise the control limit field is left empty. If one result is above five times the CRQL and the other is below, the CRQL criteria is used for control. If both results are less than the MDL, the relative percent difference is not calculated. For soil samples, the CRQL is corrected for sample weight and percent solids. If the criteria are not met, the associated samples are flagged with a "*" on the deliverable forms.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Initial Calibration

The mercury autoanalyzers are calibrated with five calibration standards and a blank at the beginning of each analytical sequence using the instrument operating conditions established by the manufacturer of the instrument. Operating instructions for the instrument are described in the instrument manual(s) located in the laboratory.

The calibration standards are prepared daily by making successive dilutions of the working standard (100ug/L). The final concentration of the prepared calibration standards is given in

the following tables:

Table I - Water

Level	Working Standard(100ug/L) mL	Final Volume mL	Final Concentration* ug/L
Blank	0	50	0
Level 1	0.1	50	0.2
Level 2	0.25	50	0.5
Level 3	0.5	50	1
Level 4	2.5	50	5
Level 5	5.0	50	10

Table II - Soil

Level	Working Standard (100 ug/L) mL	Final Volume mL	Final Concentration* ug/L
Blank	0	5.0	0
Level 1	0.1	5.0	0.2
Level 2	0.25	5.0	0.5
Level 3	0.5	5.0	1
Level 4	2.5	5.0	5
Level 5	5.0	5.0	10

**The final concentration is achieved after digestion.*

The calibration standards are digested and analyzed following the procedures given in Section 11.0. After analysis, the data system prepares a standard curve by plotting the instrument response of the calibration standards against the true value concentration and using linear regression calculates the correlation coefficient. The correlation coefficient must be greater than or equal to 0.995 and the results for each calibration level except the level equivalent to the CRQL should be within 5% of the true value. If these criteria are not met, the instrument is recalibrated.

10.2 Initial Calibration Verification (ICV)

Immediately after the instrument is calibrated, the accuracy of calibration is verified with analysis of the ICV standard. The ICV standard solution is prepared following the procedure given in Section 7.2 and digested and analyzed with the samples following the procedures given in Section 11.0. The percent recovery of the ICV must be within 80-120%. If it is not, the analysis is stopped the instrument is recalibrated and a new analytical sequence is initiated.

10.3 Continuing Calibration Verification (CCV)

To ensure the accuracy of the calibration during the analysis run, a CCV standard is analyzed at the beginning and end of each analytical run, every 10 samples or every 2 hours, whichever is more frequent. The concentration of the CCV standard should be at or near the mid point of the calibration curve and must be different than the concentration of the ICV. The CCV standard is prepared, digested and analyzed with the samples following

the procedure given in Section 11.0. The percent recovery of the CCV must be within 80-120%. If it is not, the analysis is stopped, the problem is corrected, the instrument is recalibrated and verified and all samples analyzed since the last compliant CCV are reanalyzed.

10.4 CRQL Check Standard (CRI)

To verify linearity near the CRQL, a CRI is analyzed at the beginning of each analytical run but not before the ICV and once per every 20 samples per analytical run. The CRI analysis is immediately followed by analysis of a CCV/CCB pair. The CRI standard is the same (0.2ug/L) solution used as the second level of the calibration curve. The CRI is prepared, digested and analyzed with the samples following the procedure given in Section 11.0. The percent recovery of the CRI should be within 70-130%. If it is not, the analysis is stopped, the problem is corrected the instrument is recalibrated and a new analytical sequence is initiated.

10.5 Initial and Continuing Calibration Blanks (ICB/CCB)

Calibration blanks are analyzed at each wavelength after every ICV and CCV, at a frequency of 10% or every 2 hours during the analytical sequence, whichever is more frequent. The blanks are also analyzed at the beginning of the run and after the last sample (after the last CCV). The ICB/CCB are prepared, digested and analyzed with the samples following the procedure given in Section 11.0. The absolute value of the ICB/CCB must be less than the CRQL. If it is not, the analysis is stopped, the problem is corrected, the instrument is recalibrated and verified and all samples analyzed since the last compliant ICB/CCB are reanalyzed.

10.6 Support Equipment Calibration

Check the calibration of the auto-pipettes and the top-loading balance on the day of use prior to use. Record the calibration check in the logbook designated for this purpose.

11.0 PROCEDURE

11.1 Water Sample and Standard Preparation (Manual)

For each sample, sample duplicate and sample selected as the matrix spike transfer a 50mL aliquot of sample to a labeled digestion vessel. Add 0.5mL of the working standard solution (100ug/L) to the sample selected as the matrix spike.

To prepare the preparation blank, LCS, and calibration blanks, add 50mL of reagent water to a labeled digestion vessel(s). Add 0.5mL of the working standard solution (100ug/L) to the LCS.

Prepare the calibration standards by diluting a volume of the working standard solution (100ppb) in reagent water to achieve the concentrations given in Table I. Transfer the prepared standards to labeled digestion vessel(s).

To prepare the ICV, transfer 5.0mL of the ICV working standard solution (30ppb) to a labeled digestion vessel and adjust the volume to 50mL with reagent water.

To prepare the CCV, transfer 2.5mL of the working standard solution (100ppb) to a labeled digestion vessel and adjust the volume to 50mL with reagent water.

To prepare the CRI, transfer 0.1mL of the working standard solution (100ppb) to a labeled digestion vessel and adjust the volume to 50mL with reagent water.

To each jar, add 2.5mL of concentrated sulfuric acid, 1.25mL of concentrated nitric acid, and 7.5mL of potassium permanganate solution. Allow the bottles to stand for 15 minutes. During this time, the solution should turn and remain the color purple. If after 15 minutes, the solution does not remain purple, add more potassium permanganate (~1.25mL).

Add 4mL of potassium persulfate to each jar and heat for two hours in a block digester maintained at 95°C. After the time period has elapsed, allow the jar to cool then add 3mL of hydroxylamine hydrochloride to each jar in order to reduce the excess permanganate. Swirl to ensure that any soluble residue dissolves back into solution.

Transfer each solution to individual autoanalyzer tubes in preparation for analysis.

11.2 Solid Sample and Standard Preparation (Manual)

For each sample, sample duplicate and sample selected as the matrix spike measure 0.2g of sample into a labeled digestion vessel and add 5.0mL of reagent water. Add 0.5mL of the mercury working standard solution (100ug/L) to the sample selected as the matrix spike.

To prepare the preparation blank and calibration blank(s), add 5.0mL of reagent water to a labeled digestion vessel(s). To prepare the LCS, measure 0.2g of solid LCS material into a labeled digestion vessel and add 10mL of reagent water.

Prepare the calibration standards by diluting a volume of the working standard solution (100ppb) in reagent water to achieve the concentrations given in Table II. Transfer the prepared standards to labeled digestion vessel(s).

To prepare the ICV, transfer 5.0mL of the ICV working standard solution (30ppb) to a labeled digestion vessel.

To prepare the CCV, transfer 2.5mL of the mercury working standard solution (100ppb) into a labeled digestion vessel and adjust the volume to 10mL with reagent water.

To prepare the CRI, transfer 0.1mL of the working standard solution (100ppb) to a labeled digestion vessel and adjust the volume to 50mL with reagent water.

To each jar, add 2.5mL of concentrated sulfuric acid and 1.25mL of concentrated nitric acid. Heat in a hot block digester maintained at 95°C for 2 minutes. After two minutes, remove from heat and allow the bottles to cool. Add 25mL of reagent water, 7.5mL of potassium permanganate, and 4mL of potassium persulfate and swirl to mix. Heat in a hot block



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digester maintained at 95°C for 30 minutes. Remove from heat, allow the jars to cool and add 3mL of hydroxylamine hydrochloride to reduce the excess permanganate. Swirl each jar to ensure that any soluble residue dissolves back into solution. If any sample is still purple, add additional hydroxylamine hydrochloride in 3mL increments until the purple color disappears.

Add 25mL of reagent water to each jar and then transfer the solutions to individual autoanalyzer tubes in preparation for analysis.

11.3 Instrument Set Up & Analysis

Turn on the instrument lamp, gas and pump. Allow 15 minutes for the instrument to warm up. Fill the rinse bath with 10% hydrochloric acid solution. Check all tubing connections and reset the calibration curve. Check the stannous chloride reductant reservoir and fill as necessary. Check and record the 0.2ug/L standard intensity. This value must be >2500. When applicable, record sample intensity and reference intensities and verify against previous day. Perform maintenance to increase intensities if needed.

On the PC connected to the instrument, select the autosampler template and enter the sample ids in the order of analysis. Place the samples, calibration blanks, calibration standards, and performance check standards in the position on the autosampler rack that corresponds to their assigned position in the autosampler template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin analysis.

Example Analytical Sequence:

Calibration Blank
0.2 Calibration Standard
0.5 Calibration Standard
1.0 Calibration Standard
5.0 Calibration Standard
10.0 Calibration Standard
ICV
ICB
CRI
CCV
CCB
9 Samples*
CCV
CCB
9 Samples*
CRI
CCV
CCB

**The number of samples between each CCB/CCV (10) includes preparation blanks, laboratory control samples, matrix spikes, and sample duplicates.*

After analysis is complete, review the data against the criteria given in Section 9.0 for Quality Control and Section 10.0 for Calibration and Standardization. Perform corrective action, as needed. Dilute and reanalyze any samples that exceed the linear range.

Transfer the data from the network server into the MARRS data processing software program. Route preparation log, analysis run log and associated raw data to the Inorganic Data Review department for secondary data assessment and report generation.

Note: The mercury autoanalyzer acquires the data and determines the concentration in using the equations given in Section 12.0.

11.0 CALCULATIONS

12.1 Equation 1: Water Sample Concentration

$$C_{(\mu g/L)} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{V_{samp}}$$

Where:

$\mu g/L_{dig}$ = Instrument result adjusted for dilution factors

V_{dig} = Final digestate volume

V_{samp} = Sample volume

12.2 Equation 2: Soil Sample Concentration

$$C_{(mg/Kg \text{ drywt.})} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{g_{smp}} * \frac{100}{\% \text{ solids}}$$

Where:

$\mu g/L_{dig}$ = Instrument result adjusted for dilution factors

V_{dig} = Final digestate volume

g_{smp} = Sample weight in grams

% Solids = Percent solids to nearest 0.1%

13.0 DATA ASSESSMENT, CRITERIA AND CORRECTIVE ACTION

- 13.1 All samples, standards and QC samples are reviewed against the performance criteria given in Section 9.0 for quality control and Section 10.0 for calibration and standardization. If the results do not fall within the established limits or criteria, corrective action should be performed. If corrective action is not taken or unsuccessful, the situation must be documented and reported in the project narrative. Primary review of the data is performed by the analyst(s) that performed the procedure. Secondary review is performed by a senior



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analyst or a data review analyst. All data that does not meet established criteria must be flagged with the appropriate data qualifier and noted in the project narrative

14.0 METHOD PERFORMANCE

- 14.1 A Method Detection Limit (MDL) determination is determined annually for each digestion procedure following the procedure described in 40CFR, Part 136, Appendix B and laboratory SOP LP-LB-009. The determined MDL must be less than one-half the CRQL (0.2ug/L and 0.1mg/kg for water and soil samples, respectively).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

- 15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

- 15.2 The following waste streams are produced when this method is carried out.

- Acid Waste – Satellite Container: 5 Gallon Polyethylene Container

Transfer the waste stream to the designated satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in the laboratory SOP LP-LB-0010 *Hazardous Waste*.

16.0 REVISION HISTORY

- 16.1 Table 1: changed ug/L to mL in working standard title box.
- 16.2 Table 2: changed final volume for soil calibration standards to 5.0 mL
- 16.3 Section 11.2: changed reagent water addition from 10.0 mL to 5.0 mL. Changed LCS amount from 0.1g to 0.2g.

17.0 REFERENCES

- 17.1 CLP SOW ILM05.3 For Inorganic Analyses, Multi-Media, Multi-Concentration, USEPA Office of Solid Waste and Emergency Response, February, 2004.

18.0 TABLES, DIAGRAMS & FLOWCHARTS

- 18.1 Table 1: Primary Materials Used



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18.2 Appendix A: *List of Definitions*

Table 1: Primary Materials Used, Hazards & Exposure Limit

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			



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Appendix A: List of Definitions

ANALYTE - The element or ion an analysis seeks to determine; the element of interest.

ANALYTICAL SEQUENCE - The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final CCV or CCB.

BATCH - A group of samples prepared at the same time in the same location using the same method.

BLANK - An analytical sample designed to assess specific sources of contamination.

CALIBRATION - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards.

CALIBRATION BLANK - A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation.

CALIBRATION STANDARDS - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

CONTINUING CALIBRATION VERIFICATION (CCV) - A single parameter or multiparameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples.

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) CHECK STANDARD (CRI) - A single parameter or multi-parameter standard solution prepared at the CRQL and used to verify the instrument calibration at low levels.

CONTROL LIMITS - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

DIGESTION LOG - An official record of the sample preparation (digestion).

DRY WEIGHT - The weight of a sample based on percent solids.

DUPLICATE - A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

HOLDING TIME - The elapsed time expressed in days from the date of receipt of the sample by the Contractor until the date of its analysis. Holding time = (sample analysis date - sample receipt date)



INITIAL CALIBRATION - Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

INITIAL CALIBRATION VERIFICATION (ICV) - Solution(s) prepared from stock standard solutions, metals or salts obtained from a source separate from that utilized to prepare the calibration standards.

INTERFERENTS - Substances which affect the analysis for the element of interest.

LABORATORY CONTROL SAMPLE (LCS) - A control sample of known composition. Laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the USEPA samples received.

LINEAR RANGE, LINEAR DYNAMIC RANGE - The concentration range over which the instrument response remains linear.

MATRIX - The predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, a sample matrix is either water/ aqueous or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX EFFECT - In general, the effect of particular matrix constituents.

MATRIX SPIKE - Aliquot of a sample (water/aqueous or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

METHOD DETECTION LIMIT (MDL) - The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

PERCENT DIFFERENCE (%D) - As used in this SOW and elsewhere to compare two values. The difference between the two values divided by one of the values.

PERCENT SOLIDS (%S) - The proportion of solid in a soil sample determined by drying an aliquot of the sample.

PREPARATION BLANK - An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in the SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

SAMPLE - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.



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SENSITIVITY - The slope of the analytical curve (i.e., functional relationship between instrument response and concentration).

STOCK SOLUTION - A standard solution which can be diluted to derive other standards.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report/Chain of Custody Record.

ATTACHMENT 5

STL Burlington Metals by ICP-AES



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STANDARD OPERATING PROCEDURE STL BURLINGTON

CLP SOW ILM05.3 / ICP- AES

Applicable Matrices: Waters, Soil, Sediment and Sludge

APPROVAL SIGNATURES

Christopher A. Ouellette
Laboratory Director

Date: June 20, 2006

Kirstin L. McCracken
Quality Assurance Manager

Date: June 20, 2006

William S. Cicero
Department Manager

Date: June 20, 2006

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for inductively coupled atomic plasma-atomic emission spectroscopy (ICP-AES) used to analyze water, sediment, sludge, and soil samples taken from hazardous waste sites.

1.2 This procedure is applicable to the elements listed in Table 1, Section 18.0.

2.0 SUMMARY OF METHOD

2.1 Water and soil samples are treated with acids and digested to solubilize the metals present. The digestates are analyzed for trace metals by atomic emission spectroscopy in which the samples are nebulized and the aerosol that is produced is transported to a plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed and the intensities of the lines are monitored by a photosensitive device that generates signal(s) processed by a computer. Background correction is performed to compensate for variable background contribution to the spectra. Each metal is quantified at specific wavelengths of light emitted as the electrons relax to lower energy states. The sample is analyzed by multiple integrations (2) and the average integration is converted to a concentration from a calibration curve.

2.2 This procedure is based on CLP SOW ILM05.3, Exhibit D for ICP-AES.

3.0 DEFINITIONS

3.1 A list of definitions is given in Appendix B.

4.0 INTERFERENCES

4.1 Spectral Interferences

Overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, and/or background contribution from stray light from the high concentration elements are examples of spectral interference that may cause target concentrations to be artificially high. These effects are compensated by using computer correction of the raw data by monitoring and measurement of the interfering element and/or background correction adjacent to the analyte line.

4.2 Physical Interferences

Change in viscosity and surface tension can cause significant inaccuracies especially in samples that contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump or sample dilution will minimize these interferences.



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4.3 Chemical Interferences

Chemical interferences such as molecular compound formation, ionization effects and solute vaporization effects are highly dependent on matrix type and specific analyte elements. These interferences are not typical with ICP-AES analysis but if observed, can be minimized by matrix matching, buffering the sample and careful selection of instrument operating conditions.

5.0 SAFETY

5.1. Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added. Protective clothing such as a lab coat, safety glasses and latex gloves must be worn while performing this procedure.

5.3. Primary Materials Used

Table 2, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers and part numbers are subject to change at the discretion of the laboratory.

6.1 Inductively Coupled Plasma – Atomic Emission Spectrometer (ICP-AES); Thermo Jarrell-Ash Trace ICP-AES 61E Trace or equivalent.

6.2 Block Digester: Capable of reaching 95°C.

6.3 Volumetric Pipettes: Calibrated daily.

6.4 Class A Volumetric Glassware.

6.5 Top-Loading Balance.



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- 6.6 Filter apparatus.
- 6.7 Ribbed Watch glasses.
- 6.8 Whatman No. 42 Filter Paper or equivalent.
- 6.9 Polyethylene Digestion Vessels with Volumetric Indicators.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent Water - ASTM Type II Water (ASTM D1193-77) or equivalent.

Hydrochloric Acid, Concentrated (specific gravity 1.19); reagent grade; JT Baker or equivalent

Hydrochloric Acid (1:1): Fill a carboy half full with reagent water and slowly add an equal volume of concentrated HCl. Swirl to mix.

Nitric Acid, Concentrated (specific gravity 1.41) Hydrochloric Acid; reagent grade; JT Baker or equivalent

Nitric Acid, (1:1): Fill a carboy half full with reagent water and slowly add an equal volume of concentrated HNO₃. Swirl to mix.

Hydrogen Peroxide (30%); Hydrochloric Acid; reagent grade; JT Baker or equivalent

7.2 Standards

Stock standard solutions are purchased from commercial vendors. Working, secondary dilution and mixed calibration standards are prepared from stock standards by diluting volumes of stock standards with acidified reagent water in volumetric flasks to obtain the appropriate concentration. The components and recommended concentrations for calibration, working and secondary dilution standards are given in Appendix A.

Expiration dates of multi-component prepared solutions are assigned according to expiration date of the parent standard. Standard solutions are prepared as needed or on the expiration date, whichever occurs first.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

- 8.1 All samples should be collected in glass or polyethylene containers. Water samples must be preserved with nitric acid to a pH of less than 2 immediately following collection. All samples must be iced or refrigerated to 4°C (±2°C) and maintained at that temperature until digestion.



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8.2 Dissolved Metals

For the determination of dissolved elements, the sample must be filtered through a 0.45 μm pore diameter membrane filter at the time of collection or as soon as possible. Preserve the filtrate with nitric acid to pH <2 immediately following filtration.

8.3 Samples are stored protected from light and refrigerated at 4°C (\pm 2°C) from the time of receipt until 60 days after delivery of a reconciled data package to the client. After 60 days the samples are disposed of in a manner that complies with all applicable regulations. Sample digestates are stored for 365 days after delivery of the complete, reconciled data package.

8.4 The maximum holding time for analysis is 180 days from Validated Time of Sample Receipt (VTSR).

9.0 QUALITY CONTROL

9.1 Preparation Blank (PB)

A PB is included with each digestion batch or every sample delivery group whichever is more frequent. The PB consists of reagent water and is prepared using the same reagents, in the same volumes and at the same acid concentration as the sample solution used for analysis. The PB is carried through all steps of the digestion procedure and analyzed in the same manner as samples.

The concentration of target analyte(s) in the PB must be less or equal to the CRQL. If it is not, the lowest concentration of that analyte in the associated samples must be greater than or equal to 10 times the blank concentration or the blank and associated samples must be re-digested and re-analyzed. If the concentration of the blank is below the negative CRQL, then all samples reported below 10 times the CRQL associated with the blank are re-digested and re-analyzed.

9.2 Interference Check Sample (ICS)

To verify interelement and background correction factors for all elements and for all interferents, analyze ICS solutions at the beginning and end of each analytical run, but not before the initial calibration verification (ICV) standard and at a minimum frequency of once per 20 samples per analytical run. The ICS analysis should be immediately followed by the continuing calibration blank/verification (CCV/CCB).

The ICS consists of two solutions (ICSA/ICSAB) that are analyzed consecutively starting with the ICSA. The ICSA solution includes the interferents, and the ICSAB includes the analytes mixed with the interferents.

The results of the ICSA should be within ± 2 times the CRQL of the analyte's true value or $\pm 20\%$ of the analyte's true value in the ICSA, whichever is greater assuming the true value is zero unless otherwise stated. For analytes with a CRQL less than 5000 $\mu\text{g/L}$, the ICSA results should be reported from the undiluted sample analysis. The results of the ICSAB



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solution should be within ± 2 times the CRQL of the true value or $\pm 20\%$ of the analyte's true value, whichever is greater. If the results are not within control limits, the analysis is stopped and the problem is investigated and corrected. The instrument is recalibrated and all samples analyzed since the last compliant ICSA/ICSAB are reanalyzed.

9.3 Laboratory Control Sample (LCS)

A laboratory control sample is prepared and analyzed in the same manner as samples with each digestion batch or every group of samples in a SDG, whichever is more frequent.

The percent recovery of the aqueous LCS should be within the control limits of (80-120%, except for silver and antimony). The percent recovery of the solid LCS must be within control limits given for the standard reference material used. If the results are not within the control limits, the problem is investigated, corrected, and the associated samples are re-digested and re-analyzed along with a new LCS and preparation blank.

9.4 Matrix Spike (MS)

At least one matrix spike is prepared and analyzed with each group of samples of similar matrix. The percent recovery of the matrix spike should be within the limits of 75-125%. If it is not, the results of the associated sample data is flagged with an "N" on the deliverable forms unless the sample concentration exceeds the value of spike added by a factor of four or more. In the latter event, the data is reported without qualification.

If the recovery of the matrix spike is outside control limits and the sample result does not exceed four times the concentration of spike added, a post-digestion spike is performed on the same sample that was used for the matrix spike for those elements that do not meet criteria. The sample aliquot for the post-digestion spike should be spiked at two times the indigenous level or two times the CRQL whichever is greater.

9.5 Sample Duplicate (SD)

One sample duplicate sample is prepared and analyzed from each group of samples of a similar matrix. The relative percent difference (RPD) between duplicate results should be less than or equal to 20%, when sample values are greater than or equal to five times the CRQL. If the results are less than 5 times the CRQL, a control limit of the CRQL value should be reported on the deliverable form, otherwise the control limit field is left empty. If one result is above five times the CRQL and the other is below, the CRQL criteria is used for control. If both results are less than the MDL, the relative percent difference is not calculated. For soil samples, the CRQL is corrected for sample weight and percent solids. If the criteria are not met, the associated samples are flagged with a "**" on the deliverable forms.

9.6 Serial Dilution

A serial dilution (five-fold dilution) is performed on a sample from each group of samples of a similar matrix. If the analyte concentration in the undiluted sample is sufficiently high (a factor of 50 above the MDL), the serial dilution should agree within 10% of the original



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concentration after correction for dilution. If it is not, a chemical or physical interference is suspected and the data for the associated samples is flagged with an "E" on the deliverable forms.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Calibration

Calibrate the instrument with a blank and one calibration standard for each element at the beginning of each analytical sequence using the instrument operating conditions established by the manufacturer of the instrument. Operating instructions for the instrument are described in the instrument manual(s) located in the metals laboratory.

The calibration blank and standard(s) are prepared per the instructions given in Appendix A and analyzed following the procedure given in Section 11.0. Performance check and continuing calibration standards are interspersed between blanks, QC and field samples throughout the analytical run to ensure on-going accuracy and linearity of the calibration. An example analytical sequence is given in Section 11.0

10.2 Initial Calibration Verification (ICV)

Immediately after the instrument has been calibrated, verify the accuracy of the calibration by analysis of a second source ICV standard that has been prepared in the same acid matrix as the calibration standards. The results of the ICV standard should be within the established limits of 90-110%. If it is not, stop the analysis, correct the problem, recalibrate and initiate a new analytical sequence.

10.3 Continuing Calibration Verification (CCV)

To ensure the accuracy of the calibration during the analytical run, analyze a CCV every 10 samples or every 2 hours, whichever is more frequent and at the beginning and end of each analytical run. The concentration of the elements in the CCV should be at or near the mid points of their respective calibration curve and must be different than the concentration of the ICV. The result of the CCV should be within the established limits of 90-110%. If it is not, stop the analysis, correct the problem, recalibrate and initiate a new analytical sequence.

10.4 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

To verify linearity near the CRQL, analyze a CRI at the beginning of each analytical run but not before the ICV and once per every 20 samples per analytical run. The CRI shall be run for every wavelength used for analysis, except those for Al, Ba, Ca, Fe, Mg, Na, and K. The percent recovery of the CRI should be within 70-130% (50-150% for Sb, Pb, and Tl). If it is not, stop the analysis, correct the problem, recalibrate and initiate a new analytical sequence.



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10.5 Initial and Continuing Calibration Blanks (ICB/CCB)

Analyze calibration blanks at each wavelength after every ICV and CCV, at a frequency of 10% or every 2 hours during the analytical sequence, whichever is more frequent. The blanks are also analyzed at the beginning of the run and after the last sample (after the last CCV). The absolute value of the ICB/CCB must be less than the CRQL. If it is not, stop the analysis, correct the problem, recalibrate and initiate a new analytical sequence.

10.6 Support Equipment Calibration

Check the calibration of the auto-pipettes and the top-loading balance on the day of use prior to use. Record the calibration check in the logbook designated for this purpose.

11.0 PROCEDURE

11.1 Water/Aqueous Sample Preparation (HW1) / EPA 200.7

Shake the sample to mix and transfer 100 mL to a labeled digestion vessel. Transfer two additional aliquots of the sample selected as the sample duplicate (SD) and the matrix spike (MS) to a labeled digestion vessel. Add 1mL of matrix spike solution to the matrix spike (MS) aliquot.

Note: A post digestion spike and serial dilution analyses are routinely performed on the same sample that was designated as the matrix spike. For initial analysis, the concentration of the post digestion spike standard is equivalent to 2X the CRQL for all elements. If the matrix spike recovery for any element is outside control limits, the unspiked sample result for that element does not exceed four times the concentration of spike added and if the indigenous level in the unspiked sample for that element is higher than 2X the CRQL, then the post-digestion spike must be repeated with a spike concentration equivalent to 2X the indigenous level in the unspiked sample for those elements.

To prepare the preparation blank (PB), transfer 100 mL of reagent water to a labeled digestion vessel.

To prepare the LCS, transfer 50 mL of the aqueous LCS standard to a labeled digestion vessel and dilute to 100 mL with 50 mL of reagent water.

Add 2 mL of 1:1 HNO₃ and 10mL of 1:1 HCl to each digestion vessel. Cover the digestion vessel(s) with a watch glass and place in a heated block digester maintained at a temperature of 92-95°C. Heat until the sample volume is reduced between 25 and 50 mL. Monitor the digestion to ensure that the sample does not boil and that the digestion vessel does not go dry. After digestion is complete, cool the digestate, adjust the volume to 100 mL with reagent water and allow any insoluble material to settle.

11.2 Soil Sample Preparation (HS2) SW-846 3050B

Weigh (to the nearest 0.01g) a 1.0 to 2.0 g of sample into a tared labeled digestion vessel. Weigh two additional aliquots of the sample selected as the sample duplicate (SD) and

matrix spike (MS) to individual tared, labeled digestion vessel(s). Add 2 mL of matrix spike solution to the matrix spike (MS) aliquot.

Note: A post digestion spike and serial dilution analyses are routinely performed on the same sample that was designated as the matrix spike. For initial analysis, the concentration of the post digestion spike standard is equivalent to 2X the CRQL for all elements. If the matrix spike recovery for any element is outside control limits, the unspiked sample result for that element does not exceed four times the concentration of spike added and if the indigenous level in the unspiked sample for that element is higher than 2X the CRQL, then the post-digestion spike must be repeated with a spike concentration equivalent to 2X the indigenous level in the unspiked sample for those elements.

To prepare the preparation blank (PB), weigh (to the nearest 0.01g) a 1.0 to 2.0 g of reagent water into a labeled digestion vessel.

To prepare the LCS, weigh (to the nearest 0.01g) a 1.0 to 2.0 g of solid LCS standard material into a labeled digestion vessel.

Add 10ml of 1:1 HNO₃ to each digestion vessel. Cover with a watch glass and place in a heated block digester maintained at a temperature of 92-95°C. Heat the sample for 10 minutes without boiling. Allow the sample to cool, and 5mL of concentrated HNO₃. Replace the watch glass and reflux for 30 minutes. Add an additional 5ml of concentrated HNO₃ and reflux. Repeat this step until no brown fumes are given off by the sample indicating that oxidation is complete.

Allow the sample to cool and add 2 mL of reagent water and 3 mL of 30% hydrogen peroxide. Replace the watch glass and return the digestion vessel to the block digester. Warm to initiate the peroxide action and continue to heat until the effervescence subsides. Allow the sample to cool. Continue to add 30%hydrogen peroxide in 1mL aliquots with warming until the effervescence is minimal or until sample appearance remains unchanged.

Do not add more than 10mL of 30% hydrogen peroxide. Add 10mL of concentrated HCl to each digestion vessel. Replace the watch glass and return the digestion vessel to the block digester. Heat for 10 minutes. After digestion is complete, cool the digestate, adjust the volume to 100mL with reagent water and allow any insoluble material to settle.

11.3 Standards Preparation

Prepare the mixed calibration standards and on-going performance check standards (ICV, CCV, CRI, ICSA, ICSAB) using the formulation given in Appendix A. Transfer approximately 25 mL of each prepared standard to individual, labeled autosampler tubes.

Prepare each calibration blank by transferring approximately 25 mL of 5% hydrochloric acid/2% nitric acid solution into individual, labeled autosampler tubes.

11.4 ICP-AES Analysis

Set up and profile the instrument using the operating conditions recommended by the manufacturer. Allow sufficient time for the instrument to become thermally stable prior to



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calibration.

For each sample, transfer approximately 8mL of digestate to individual autosampler tubes. Prepare the serial dilution by transferring 1mL of parent sample and 4 mL of the 5% hydrochloric acid/2% nitric acid solution to an autosampler tube.

Prepare a post digestion spike by transferring 0.8 mL of the post-spike solution and 7.2 mL of parent sample to an autosampler tube. To prepare the serial dilution, transfer 1 mL of the sample into an autosampler tube and add 4 mL of reagent water.

On the PC connected to the instrument, select the autosampler template and enter the sample ids in the order of analysis. Place the samples, serial dilution, post-digestion spike, calibration blanks, mixed calibration standards, and performance check standards in the position on the autosampler rack that corresponds to their assigned position in the autosampler template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin analysis.

An example analytical sequence is given below:

Calibration Blank
Calibration Standard #7
Calibration Standard #8
Calibration Standard #4
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
10 Samples*
CCV
CCB
7 Samples*
CRI
ICSA
ICSAB
CCV
CCB

**The number of samples between each CCB/CCV (10) includes preparation blanks, laboratory control samples, matrix spikes, sample duplicates, serial dilutions and the post digestion spike.*

After analysis is complete, review the data against the criteria given in Section 9.0 for Quality Control and Section 10.0 for Calibration and Standardization. Perform corrective action, as needed. Dilute and reanalyze any samples that exceed the linear range and perform post digestion spikes as necessary.

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Transfer the data from the network server into the MARRS data processing software program. Route preparation log, analysis run log and associated raw data to the Inorganic Data Review department for secondary data assessment and report generation.

Note: The data processing software is configured to acquire two replicate exposures for all QC and sample analyses and to use the average result of multiple exposures for standardization. The data system determines the concentration using the equations given in Section 12.0.

12.0 CALCULATIONS

Unless otherwise specified, calculations are performed using data analysis software.

12.1 Equation 1: Water Sample Concentration

$$C_{(ug/L)} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{V_{samp}}$$

Where:

$\mu g/L_{dig}$ = ICP result including all dilution factors

V_{dig} = final digestate volume in mLs

V_{samp} = sample volume in mLs

12.2 Equation 2: Soil/Sediment Sample Concentration

Where:

$\mu g/L_{dig}$ = ICP result including all dilution factors

V_{dig} = final digestate volume in Liters

g_{samp} = sample weight in grams

13.0 DATA ASSESSMENT, CRITERIA AND CORRECTIVE ACTION

- 13.1 Primary review is performed and documented by the analyst that performed the digestion or analysis procedure. All data undergoes secondary review by a senior analyst or data review analyst. The review ensures that all data is reviewed against the performance criteria given in Section 9.0 for quality control and Section 10.0 for calibration and standardization. If the results are not within the established limits or criteria, corrective action is performed. If corrective action is not taken or is unsuccessful, the situation is documented and reported in the project narrative. All data that does not meet established criteria is flagged with the



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appropriate data qualifier and noted in the project narrative.

14.0 METHOD PERFORMANCE

14.1 Method Detection Limit (MDL) determinations are determined annually on each instrument for each digestion procedure, and non-prepared analyses following the procedure described in 40CFR, Part 136, Appendix B and laboratory SOP LP-LB-009. The determined MDL must be less than one-half the CRQL. The non-digested MDL is used to determine data qualifiers for the results of the non-prepared samples and QC analyses.

14.2 Interelement Corrections

Interelement correction factors are determined quarterly by the analysis of a single element standard for each element at an environmentally representative concentration. For each of those analyses the presence of a positive or negative value whose absolute value for any other element exceeds the CRQL is documented. A "K" factor is determined by dividing the concentration observed of a given element by the concentration of the interfering element and is entered into the software system. Following this, the ICSA solution is analyzed and the process is repeated for any elements not present in the solution that exhibit a positive or negative value. When the procedure is complete, all "K" values are considered final if the ICSA analysis meets the acceptance criteria for the ICSA analysis.

14.3 Linear Range Analysis

Linear range analysis is determined quarterly by the analysis of a multi-component or single element standard for each element. The highest concentration recovered within 5% of its true value determines the linear range for the instrument.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Acidic Waste – Satellite Container: 5 Gallon Polyethylene Container

Transfer the waste stream to the designated satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in the laboratory SOP LP-LB-0010 *Hazardous Waste*.

16.0 REVISION HISTORY

16.1 Title Page: Updated to current management team.



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- 16.2 Section 5.0: Updated safety information.
- 16.3 Section 6.0: Updated instrument descriptions.
- 16.4 Section 7.0: Updated reagent information.
- 16.5 Section 15.0: Updated pollution prevention and waste management to reflect current practice and added waste stream information.
- 16.6 Section 18.0: Added Table 2.

17.0 REFERENCES

- 16.1 CLP SOW ILM05.3 For Inorganic Analyses, Multi-Media, Multi-Concentration, USEPA Office of Solid Waste and Emergency Response, February, 2004.

18.0 TABLES, DIAGRAMS & FLOWCHARTS

- 18.1 Table 1: TCL List, CRQLs and Analyte Wavelengths Used by Instrument
- 18.2 Table 2: Primary Materials Used
- 18.3 Appendix A: Standard Preparation Tables
- 18.4 Appendix B: List of Definitions

Table 1: Target Analyte List (TCL), CRQLs and Analyte Wavelengths Used by Instrument

Analyte	CAS No.	CRQL		Analyte Wavelengths Used (nm) by Instrument		
		Water (ug/L)	Soil (mg/kg)	TJA Trace ICP4	TJA Trace ICP5	TJA Trace ICP6
Aluminum	7429-90-5	200	20	308.215	237.313	308.215
Antimony	7440-36-0	60	6	206.838	206.838	206.838
Arsenic	7440-38-2	10	1	189.042	189.042	189.042
Barium	7440-39-3	200	20	493.409	493.409	493.409
Beryllium	7440-41-7	5	0.5	313.042	313.042	313.042
Cadmium	7440-43-9	5	0.5	226.502	226.501	226.502
Calcium	7440-70-2	5000	500	317.933	317.933	317.933
Chromium	7440-47-3	10	1	267.716	267.716	267.716
Cobalt	7440-48-4	50	5	228.616	228.611	228.616
Copper	7440-50-8	25	2.5	324.754	324.754	324.753
Iron	7439-89-6	100	10	271.441	271.441	271.441
Lead	7439-92-1	10	1	220.353	220.353	220.353
Magnesium	7439-95-4	5000	500	279.078	279.079	279.078
Manganese	7439-96-5	15	1.5	257.610	294.920	257.610
Nickel	7440-02-0	40	4	231.604	231.601	202.030
Potassium	7440-09-7	5000	500	766.491	776.491	766.491
Selenium	7782-49-2	35	3.5	196.026	196.026	196.026
Silver	7440-22-4	10	1	328.068	328.068	328.068
Sodium	7440-23-5	5000	500	330.232	330.232	330.232
Thallium	7440-28-0	25	2.5	190.864	421.552	190.864
Vanadium	7440-62-2	50	5	292.402	292.402	292.402
Zinc	7440-66-6	60	6	231.856	213.851	206.200

Table 2: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm- STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			



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Appendix A: Standard Preparation Tables

Unless otherwise specified all standards are prepared with a 5% HCl / 2% HNO₃ solution.

Mixed Element Calibration Standards

Calibration Standard #7

Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
500ppm Calcium	200	2000	50000
500ppm Potassium			50000
500ppm Sodium			50000
500ppm Magnesium			50000
500ppm Aluminum			50000
500ppm Iron			50000

Calibration Standard #8

Single Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
1000ppm Arsenic	1.0	2000	500
1000ppm Selenium	1.0	2000	500
1000ppm Thallium	1.0	2000	500
1000ppm Antimony	1.0	2000	500
1000ppm Lead	2.0	2000	1000
1000ppm Tin	2.0	2000	1000
1000ppm Strontium	2.0	2000	1000
1000ppm Titanium	2.0	2000	1000

Calibration Standard #4

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
Silver	1.0	2000	500
Phosphorus	2.0		1000
Silicon	10		5000
Boron	20 X-AQU-5	2000	1000
Barium			1000
Beryllium			500
Cadmium			500
Cobalt			1000
Chromium			1000
Copper			1000
Manganese			1000
Molybdenum			1000
Nickel			1000
Vanadium			1000
Zinc			1000



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CRI Standards

CRI Working Standard Solution

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
CLP-AES-CRQL Stock	20	2000	See below
1000ppm Sn	0.08	2000	40
1000ppm B	0.4	2000	200
1000ppm Mo	0.04	2000	20
1000ppm Sr	0.08	2000	40
1000ppm P	1.0	2000	500
1000ppm Ti	0.08	2000	40
1000ppm Si	0.4	2000	200

CLP-AES-CRQL: Stock Standard Solution

Stock Standard Used -- Element	Initial Standard Concentration (ug/mL)	Final Standard Concentration (ug/L)
Ca Mg K Na	500	5000
Al Ba	20	200
Fe	10	100
Sb Zn	6	60
Co V	5	50
Ni	4	40
Se	3.5	35
Cu Ti	2.5	25
As Mn	1.5	15
Cr Pb Ag	1	10
Be Cd	0.5	5



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Interference Check (ICS) Solutions

ICSA Working Standard Solution

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
CLPP-ICS-A Stock	100	2000	See below

CLPP-ICS-A Stock Standard Solution

Stock Standard Used – Element	Initial Standard Concentration (mg/L)	Final Standard Concentration (mg/L)
Al Ca Mg	5000	250
Fe	2000	100

ICSAB Working Standard Solution

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
CLPP-ICS-A Stock	100	2000	See above
CLPP-ICS-B4 Stock	20	2000	See below
1000ppm B	3	2000	1500
1000ppm Sn	3	2000	1500
1000ppm Mo	2	2000	1000
1000ppm Si	2	2000	1000
1000ppm P	1	2000	500
1000ppm Ti	1	2000	500
1000ppm Sr	0.5	2000	250

CLPP-ICS-B4: Stock Standard Solution

Stock Standard Used – Element	Initial Standard Concentration (mg/L)	Final Standard Concentration (ug/L)
Cd Ni Zn	100	1000
Sb	60	600
Ba Be Co Cr Cu Mn V	50	500
Ag	20	200
As Tl	10	100
Pb Se	5	50



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Initial Calibration Verification (ICV)

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
100ppm Aluminum	10 AT-2	1000	26,000
100ppm Lead			1000
50ppm Barium			500
50ppm Beryllium			500
50ppm Boron			500
50ppm Cadmium			500
50ppm Chromium			500
50ppm Cobalt			500
50ppm Iron			25,500
50ppm Manganese			500
50ppm Nickel			500
50ppm Silver			500
50ppm Strontium			500
50ppm Vanadium			500
50ppm Zinc			500
50ppm Copper			500
500ppm Aluminum	50 AT-3	1000	See Above
500ppm Calcium			25,000
500ppm Iron			See Above
500ppm Magnesium			25,000
500ppm Potassium			25,000
500ppm Sodium			25,000
1000ppm Molybdenum	0.5		500
1000ppm Antimony	0.25		250
1000ppm Thallium	0.25		250
1000ppm Selenium	0.25		250
1000ppm Arsenic	0.25		250
1000ppm Tin	0.25		250
1000ppm Titanium	0.5		500
1000ppm Phosphorus	0.5		500
1000ppm Silicon	0.25		250

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Continuing Calibration Verification (CCV)

CCV Working Standard Solution

CCV Working Standard Solution				
Single or Multi-Element Stock Standard	Stock Standard Concentration (ppm)	Volume Stock (mL)	Volume Prepared (mL)	Final Concentration (ug/L)
Silver	Cal Standard #4	200	1000	100
Phosphorus				200
Silicon				1000
Boron				700
Barium				200
Beryllium				100
Cadmium				100
Cobalt				200
Chromium				200
Copper				200
Manganese				200
Molybdenum				200
Nickel				200
Vanadium				200
Zinc				200
Calcium	Cal Standard #7	600		30,200
Potassium				30,200
Sodium				30,200
Magnesium				30,200
Aluminum				30,200
Iron				30,200
Arsenic	Cal Standard #8	200		100
Selenium				100
Thallium				100
Antimony				300
Lead				400
Tin				200
Titanium				400
Strontium				300
Boron	1000 ppm	0.5		See Above



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Matrix Spike Solution

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)
10000 ppm Al	10	500	200
1000 ppm Sb	5	500	10
1000 ppm As	2	500	4
1000 ppm Ba	100	500	200
1000 ppm Be	2.5	500	5
1000 ppm Cd	2.5	500	5
1000 ppm Cr	10	500	20
1000 ppm Co	25	500	50
1000 ppm Cu	12.5	500	25
10000 ppm Fe	5	500	100
1000 ppm Pb	1	500	2
1000 ppm Mn	25	500	50
1000 ppm Ni	25	500	50
1000 ppm Se	2.5	500	5
1000 ppm Tl	2.5	500	5
1000 ppm V	25	500	50
1000 ppm Zn	25	500	50
1000 ppm B	25	500	50
1000 ppm Mo	25	500	50
1000 ppm Sn	25	500	50
1000 ppm Si	25	500	50
1000 ppm P	25	500	50
1000 ppm Ti	25	500	50
1000 ppm Ag	2.5	500	5
1000 ppm Sr	25	500	50

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Aqueous LCS Solution

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)
**AT - 2	40	2000	See below
***AT - 3	400	2000	See below
1000ppm Sb	8	2000	4
1000ppm As	4	2000	2
1000ppm Sn	4	2000	2
1000ppm Mo	4	2000	2
1000ppm Se	2	2000	1
1000ppm Tl	2	2000	1
1000ppm Sr	4	2000	2
1000ppm Si	4	2000	2
1000ppm P	4	2000	2
1000ppm Ti	4	2000	2
****LCSWF	20	2000	See below

** AT - 2

Stock Standard Used – Element	Initial Concentration (mg/L)	Final Standard Concentration (mg/L)
Al and Pb	100	2
Ba, Be, B, Cd, Cr, Co, Fe Mn, Ni, Ag, Sr, V and Zn	50	1

*** AT - 3

Stock Standard Used – Element	Initial Standard Concentration (mg/L)	Final Standard Concentration (mg/L)
Al, Ca, Fe, Mg, K and Na	500	100

** **LCSWF

Stock Standard Used – Element	Initial Standard Concentration (mg/L)	Final Standard Concentration (mg/L)
As and Tl	10	0.1
Cd and Se	5	0.05
Pb	3	0.03



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Appendix B: List of Definitions

ANALYTE - The element or ion an analysis seeks to determine; the element of interest.

ANALYTICAL SEQUENCE - The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final CCV or CCB.

BACKGROUND CORRECTION - A technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

BATCH - A group of samples prepared at the same time in the same location using the same method.

BLANK - An analytical sample designed to assess specific sources of contamination.

CALIBRATION - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards.

CALIBRATION BLANK - A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation.

CALIBRATION STANDARDS - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

CONTINUING CALIBRATION VERIFICATION (CCV) - A single parameter or multiparameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples.

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) CHECK STANDARD (CRI) - A single parameter or multi-parameter standard solution prepared at the CRQL and used to verify the instrument calibration at low levels.

CONTROL LIMITS - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

DISSOLVED METALS - Analyte elements in a water/aqueous sample which will pass through a 0.45 micrometer (μm) filter.



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DRY WEIGHT - The weight of a sample based on percent solids. The weight after drying in an oven.

DUPLICATE - A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

HOLDING TIME - The elapsed time expressed in days from the date of receipt of the sample by the Contractor until the date of its analysis.

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY (ICP-AES) – A technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

INITIAL CALIBRATION - Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

INITIAL CALIBRATION VERIFICATION (ICV) - Solution(s) prepared from stock standard solutions, metals or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration.

INTERFERENCE CHECK SAMPLE - A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

INTERFERENTS - Substances which affect the analysis for the element of interest.

LABORATORY CONTROL SAMPLE (LCS) - A control sample of known composition.

LINEAR RANGE, LINEAR DYNAMIC RANGE - The concentration range over which the instrument response remains linear.

MATRIX - The predominant material of which the sample to be analyzed is composed.

MATRIX EFFECT - In general, the effect of particular matrix constituents.

MATRIX SPIKE - Aliquot of a sample (water/aqueous or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.



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METHOD DETECTION LIMIT (MDL) - The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

PERCENT DIFFERENCE (%D) - The difference between the two values divided by one of the values.

PERCENT SOLIDS (%S) - The proportion of solid in a soil sample determined by drying an aliquot of the sample.

PREPARATION BLANK - An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

RELATIVE PERCENT DIFFERENCE (RPD) - The relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

SAMPLE - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SENSITIVITY - The slope of the analytical curve (i.e., functional relationship between instrument response and concentration).

SERIAL DILUTION - The dilution of a sample by a factor of five. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

STOCK SOLUTION - A standard solution which can be diluted to derive other standards.

TARGET ANALYTE LIST (TAL) - A list of Inorganic Analytes (metals and cyanide) as designated in Exhibit C.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report/Chain of Custody Record.

ATTACHMENT 6

STL Burlington Metals by ICP-MS

STL BURLINGTON
SOP CHANGE-IN-PROGRESS ATTACHMENT (CIPA)

SOP Title: CLP SOW ILM05.3 / ICP- MS
SOP No: LM-MI-ILM05.3-MS
Revision: 1
Date Effective: 07/27/06
CIPA Date Effective: 10/27/06

Change Approved By:

QA Manager: Kirstin McCracken **Date:** October 27, 2006
Kirstin McCracken

Inorganic Manager: William S. Cicero **Date:** October 27, 2006
William S. Cicero

<p>The following revisions or additions in BOLD TEXT were made to the referenced SOP. These changes were implemented on the CIPA Date Effective indicated above.</p>

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Working Calibration Standard Solutions

Element	Intermediate Calibration Standard (mg/L)	Calibration Level 3			Calibration Level 2 (Prepared from Cal 3)			Calibration Level 1 (Prepared from Cal 3)		
		Initial Volume (mL)	Final Volume (mL)	Final Concentration (mg/L) (ug/L)	Initial Volume (mL)	Final Volume (mL)	Final Concentration (mg/L) (ug/L)	Initial Volume (mL)	Final Volume (mL)	Final Concentration (mg/L) (ug/L)
Al	40	25	1000	1000	100	500	200	20	500	40
Sb	20	25	1000	500	100	500	100	20	500	20
As	2.0	25	1000	50	100	500	10	20	500	2.0
Ba	100	25	1000	2500	100	500	500	20	500	100
Be	2.0	25	1000	50	100	500	10	20	500	2.0
B	20	25	1000	500	100	500	100	20	500	20
Cd	2.0	25	1000	50	100	500	10	20	500	2.0
Ca	1000	25	1000	25000	100	500	5000	20	500	1000
Cr	4.0	25	1000	100	100	500	20	20	500	4.0
Co	10	25	1000	250	100	500	50	20	500	10
Cu	20	25	1000	500	100	500	100	20	500	20
Fe	200	25	1000	5000	100	500	1000	20	500	200
Pb	2.0	25	1000	50	100	500	10	20	500	2.0
Mg	1000	25	1000	25000	100	500	5000	20	500	1000
Mn	4.0	25	1000	100	100	500	20	20	500	4.0
Mo	20	25	1000	500	100	500	100	20	500	20
Ni	4.0	25	1000	100	100	500	20	20	500	4.0
K	1000	25	1000	25000	100	500	5000	20	500	1000
Se	2.0	25	1000	50	100	500	10	20	500	2.0
Ag	2.0	25	1000	50	100	500	10	20	500	2.0
Na	1000	25	1000	25000	100	500	5000	20	500	1000
Tl	2.0	25	1000	50	100	500	10	20	500	2.0
V	4.0	25	1000	100	100	500	20	20	500	4.0
Zn	20	25	1000	500	100	500	100	20	500	20

Solution: 2% Nitric Acid

Note: Working calibration standards Level 1 and Level 2 are secondary dilutions of calibration standard level 3, which is prepared from the intermediate calibration standard solution. The working calibration standards must be assigned an expiration date of 14 days from date of preparation or in accordance with the expiration date of the parent standard, whichever occurs first.



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STANDARD OPERATING PROCEDURE STL BURLINGTON

CLP SOW ILM05.3 / ICP- MS

Applicable Matrices: Waters

APPROVAL SIGNATURES

Christopher A. Ouellette
Laboratory Director

Date: June 20, 2006

Kirstin L. McCracken
Quality Assurance Manager

Date: June 20, 2006

William S. Cicero
Department Manager

Date: June 20, 2006

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R2-0008210



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1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes the laboratory procedure for the use of inductively coupled atomic plasma-mass spectrometry (ICP-MS) used to analyze water and aqueous samples taken from hazardous waste sites.
- 1.2 This procedure is applicable to the elements listed in Table 1, Section 18.0.

2.0 SUMMARY OF METHOD

- 2.1 A sample submitted for analysis is acid digested, nebulized and transported by argon gas to a plasma torch. The ions produced are introduced via direct interface into a mass spectrometer and sorted according to their mass-to-charge ratios, quantified using an electron multiplier and processed by a data processing system.
- 2.2 This procedure is based on CLP SOW ILM05.3, Exhibit D.

3.0 DEFINITIONS

- 3.1 A list of definitions is given in Appendix B.

4.0 INTERFERENCES

4.1 Isobaric Elemental Interferences

Isobaric elemental interferences are caused by isotopes of different elements that form singly or doubly charged ions of the same nominal mass-to-charge ratio and cannot be resolved by the mass spectrometer. Correction for this interference is handled by the data system and is accomplished by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest.

4.2 Abundance Sensitivity

Abundance sensitivity is a property that defines the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and mass filter operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize occurrence.

4.3 Isobaric Polyatomic Ion Interference

Isobaric Polyatomic Ion Interference is caused by ions consisting of more than one atom, which have the same mass-to-charge ratio as the isotope of interest and cannot be resolved by the mass spectrometer. These interferences should be recognized, and when they cannot be avoided by the selection of alternative isotopes, appropriate

corrections should be made to the data. Instrument operating parameters should be set to conditions that will minimize this phenomenon.

4.4 Physical Interference

Physical interferences such as a change in viscosity, surface tension and dissolved solids are known to affect instrument responses to certain samples. These physical effects can be reduced by dilution, matrix matching, as well as not allowing dissolved solids within the samples to exceed 0.2% (w/v).

4.5 Memory Interferences

Memory interferences result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. The minimum rinse time is set at 120 seconds. Memory interference can also be identified by monitoring the three replicate integrations that are used for data acquisition.

4.6 Polyatomic Interference

Nitric acid is preferred for ICP-MS in order to minimize polyatomic interferences known in the presence of the chloride ion. Hydrochloric acid is required to maintain stability in solutions containing antimony and silver. Corrections for chloride polyatomic interferences are applied to data regardless of whether hydrochloric acid is used in digestion, as the chloride ion is common in environmental samples.

5.0 SAFETY

5.1. Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added. Protective clothing such as a lab coat, safety glasses and latex gloves must be worn while performing this procedure.

5.3. Primary Materials Used

Table 2, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.



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6.0 EQUIPMENT AND SUPPLIES

- 6.1 Inductively Coupled Plasma Mass Spectrometer: Thermo Electron X series
- 6.2 Variable Speed Peristaltic Pump
- 6.3 Water Cooled Spray Chamber
- 6.4 Block Digester: Capable of reaching 95°C
- 6.5 Volumetric Pipettes: Calibrated daily
- 6.6 Class A Volumetric Glassware
- 6.7 Top-Loading Balance
- 6.8 Filter apparatus
- 6.9 Ribbed Watch glasses
- 6.10 Whatman No. 42 Filter Paper or equivalent
- 6.11 Polyethylene Digestion Vessels with Volumetric Indicators.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent Water - ASTM Type II Water (ASTM D1193-77) or equivalent.

Hydrochloric Acid, Concentrated (specific gravity 1.19); reagent grade; JT Baker or equivalent

Hydrochloric Acid (1:1): Fill a carboy half full with reagent water and slowly add an equal volume of concentrated HCl. Swirl to mix.

Nitric Acid, Concentrated (specific gravity 1.41); reagent grade; JT Baker or equivalent

Nitric Acid, (1:1): Fill a carboy half full with reagent water and slowly add an equal volume of concentrated HNO₃. Swirl to mix.

Nitric Acid Solution (2% v/v): Slowly add 20mL of concentrated nitric acid to 980mL of reagent water.

Hydrogen Peroxide (30%); reagent grade; JT Baker or equivalent



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7.2 Standards

Due to the sensitivity of the ICP/MS, all reagents must be high-purity, whenever possible. Stock standard solutions are purchased from commercial vendors. Working, secondary dilution and mixed calibration standards are prepared from stock standards by diluting volumes of stock standards with acidified reagent water in volumetric flasks to obtain the appropriate concentration. The components and recommended concentrations for calibration, working and secondary dilution standards are given in Appendix A.

The working calibration standards are assigned an expiration date of 14 days from date of preparation, or the date of expiration of the parent standard, whichever occurs first. Expiration dates for the other multi-component prepared solutions are assigned according to expiration date of the parent standard and prepared as needed or on the expiration date, whichever occurs first.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT & STORAGE

- 8.1 All samples should be collected in glass or polyethylene containers. Aqueous samples must be preserved with nitric acid to a pH of less than 2. Immediately following collection, all samples must be iced or refrigerated to 4°C ($\pm 2^\circ\text{C}$) and maintained at that temperature until digestion.

For the determination of dissolved elements, the unpreserved sample must be filtered through a 0.45 μm pore diameter membrane filter at the time of collection and preserved with nitric acid to pH <2 immediately following filtration.

- 8.2 Samples are stored protected from light and refrigerated at 4°C ($\pm 2^\circ\text{C}$) from the time of receipt until 60 days after delivery of a reconciled data package to the client. After 60 days the samples are disposed of in a manner that complies with all applicable regulations. Sample digestates are stored for 365 days after delivery of the complete, reconciled data package.
- 8.3 The maximum holding time for analysis is 180 days from Validated Time of Sample Receipt (VTSR).

9.0 QUALITY CONTROL

- 9.1 Preparation Blank (PB)

A PB is included with each digestion batch or every sample delivery group whichever is more frequent. The PB consists of reagent water and is prepared using the same reagents, in the same volumes and at the same acid concentration as the sample solution used for analysis. The PB is carried through all steps of the digestion procedure and analyzed in the same manner as samples.

The concentration of target analyte(s) in the PB must be less or equal to the CRQL. If it is not, the lowest concentration of that analyte in the associated samples must be

greater than or equal to 10 times the blank concentration or the blank and associated samples must be re-digested and re-analyzed. If the concentration of the blank is below the negative CRQL, then all samples reported below 10 times the CRQL associated with the blank are re-digested and re-analyzed.

9.2 Interference Check Sample (ICS)

To verify interelement and background correction factors for all elements and for all interferences, ICS solutions are analyzed at the beginning of each analytical run, but not before the initial calibration verification (ICV) standard. The ICS analysis should be immediately followed by the continuing calibration blank/verification (CCV/CCB).

The ICS consists of two solutions (ICSA/ICSAB) that are analyzed consecutively starting with the ICSA. The ICSA solution includes the interferences, and the ICSAB includes the analytes mixed with the interferences.

The results of the ICSA should be within ± 3 times the CRQL of the analyte's true value or $\pm 20\%$ of the analyte's true value in the ICSA, whichever is greater assuming the true value is zero unless otherwise stated. The results of the ICSAB solution should be within ± 3 times the CRQL of the true value or $\pm 20\%$ of the analyte's true value, whichever is greater. If the results are not within control limits, the analysis is stopped and the problem is investigated and corrected. The instrument is recalibrated and all samples analyzed since the last compliant ICSA/ICSAB are reanalyzed.

9.3 Laboratory Control Sample (LCS)

A laboratory control sample is prepared and analyzed in the same manner as samples with each digestion batch or every group of samples in a SDG, whichever is more frequent.

The percent recovery of the aqueous LCS should be within the control limits of 80-120%. If the results are not within the control limits, the problem is investigated, corrected, and the associated samples are re-digested and re-analyzed along with a new LCS and preparation blank.

9.4 Matrix Spike (MS)

At least one matrix spike is prepared and analyzed with each group of samples of similar matrix. The percent recovery of the matrix spike should be within the limits of 75-125%. If it is not, the results of the associated sample data is flagged with an "N" on the deliverable forms unless the sample concentration exceeds the value of spike added by a factor of four or more. In the latter event, the data is reported without qualification.

If the recovery of the matrix spike is outside control limits and the sample result does not exceed four times the concentration of spike added, a post-digestion spike is performed on the same sample that was used for the matrix spike for those elements that do not meet criteria. The sample aliquot for the post-digestion spike should be spiked at two times the indigenous level or two times the CRQL whichever is greater.



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9.5 Sample Duplicate (SD)

One sample duplicate sample is prepared and analyzed from each group of samples of a similar matrix. The relative percent difference (RPD) between duplicate results should be less than or equal to 20%, when sample values are greater than or equal to five times the CRQL. If the results are less than 5 times the CRQL, a control limit of the CRQL value should be reported on the deliverable form, otherwise the control limit field is left empty. If one result is above five times the CRQL and the other is below, the CRQL criteria is used for control. If both results are less than the MDL, the relative percent difference is not calculated. For soil samples, the CRQL is corrected for sample weight and percent solids. If the criteria are not met, the associated samples are flagged with a "*" on the deliverable forms.

9.6 Serial Dilution

A serial dilution (five-fold dilution) is performed on a sample from each group of samples of a similar matrix. If the analyte concentration in the undiluted sample is sufficiently high (a factor of 50 above the MDL), the serial dilution should agree within 10% of the original concentration after correction for dilution. If it is not, a chemical or physical interference is suspected and the data for the associated samples is flagged with an "E" on the deliverable forms.

9.7 Internal Standard

Internal standards are used to correct for instrument drift and physical interference. A list of internal standards used for this procedure is given in Table 2. For full range mass scans, a minimum of three internal standards that bracket the masses of the analyte should be used. Internal standards are added to all samples, standards, and blanks in the same manner and at the same concentration. The absolute response of any one internal standard must not deviate more than 60 to 125% of the original response in the calibration blank.

10.0 CALIBRATION & STANDARDIZATION

10.1 Pre-Calibration Routine & Tune Standard

The instrument is calibrated prior to each analytical run. Prior to calibration, the instrument is allowed to become stable following the manufacturer's instrument operating parameters. Mass calibration and resolution routines are performed to bring the peak width within 0.75amu at 10% peak height and to within 0.1amu over the range of 6 to 210amu. Detector-mass calibration and cross calibration are re-determined as needed or whenever a major change (i.e. installation of new detector) in instrumentation is implemented.

To demonstrate instrument stability and precision, a tune standard is analyzed five times consecutively. If the peak width at 10% peak height is not within 0.75amu for each isotope, the mass calibration is not within 0.1amu over the range of 6 to 210amu, or the

percent relative standard deviation of the absolute signals for all analytes in the tune standard exceeds 5%, the tune standard is immediately reanalyzed to ensure the instrument has reached thermal stability. If repeat failure the problem is investigated, and the tune check is repeated prior to calibration.

10.2 Instrument Calibration

After analysis of the tune standard, the instrument is calibrated with a blank and three standards. The data system acquires a minimum of three replicate integrations and the average of the integrations is used for instrument calibration and quantitation. The correlation coefficient for the curve must be >0.995 for each analyte of interest in order to proceed with analysis. Each analytical sequence uses the instrument operating conditions established by the manufacturer of the instrument. Operating instructions for the instrument are described in the instrument manual(s) located in the metals laboratory.

The calibration blank and standard(s) are prepared per the instructions given in Appendix A and analyzed following the procedure given in Section 11.0. Performance check and continuing calibration standards are interspersed between blanks, QC and field samples throughout the analytical run to ensure on-going accuracy and linearity of the calibration. An example analytical sequence is given in Section 11.0

10.3 Initial Calibration Verification (ICV)

Immediately after the instrument has been calibrated, the accuracy of the calibration is verified by analysis of a second source ICV standard that has been prepared in the same acid matrix as the calibration standards. The results of the ICV standard should be within the established limits of 90-110%. If it is not, the analysis is stopped the instrument is recalibrated and a new analytical sequence is initiated.

10.4 Continuing Calibration Verification (CCV)

To ensure the accuracy of the calibration during the analytical run, a CCV is analyzed every 10 samples or every 2 hours, whichever is more frequent and at the beginning and end of each analytical run. The concentration of the elements in the CCV should be at or near the mid points of their respective calibration curve and must be different than the concentration of the ICV. The result of the CCV should be within the established limits of 90-110%. If it is not, the analysis is stopped, the problem is corrected the instrument is recalibrated and a new analytical sequence is initiated.

10.5 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

To verify linearity near the CRQL, a CRI is analyzed at the beginning and end of each analytical run but not before the ICV. The CRI is also analyzed at a frequency of not less than once per 20 analytical samples per analysis run. The CRI shall be run for every isotope used for analysis. The percent recovery of the CRI should be within 70-130% (50-150% for Co, Mn, and Zn). If it is not, the analysis is stopped, the problem is corrected the instrument is recalibrated and a new analytical sequence is initiated.

10.6 Initial and Continuing Calibration Blanks (ICB/CCB)

Calibration blanks are analyzed at each isotope after every ICV and CCV, at a frequency of 10% or every 2 hours during the analytical sequence, whichever is more frequent. The blanks are also analyzed at the beginning of the run and after the last sample (after the last CCV). The absolute value of the ICB/CCB must be less than the CRQL. If it is not, the problem is corrected the instrument is recalibrated and a new analytical sequence is initiated.

10.7 Support Equipment Calibration

Check the calibration of the auto-pipettes and the top-loading balance on the day of use prior to use. Record the calibration check in the logbook designated for this purpose.

11.0 PROCEDURE

11.1 Water/Aqueous Sample Preparation (HW3)

Shake the sample to mix and transfer 100mL to a labeled digestion vessel. Transfer an additional aliquot each of the sample selected as the sample duplicate (SD) and the matrix spike (MS) to a labeled digestion vessel(s). Add 1mL of Working Stock Matrix Spike Standard Solution to the matrix spike (MS) aliquot.

Note: A post digestion spike and serial dilution analyses are routinely performed on the same sample that was designated as the matrix spike. For initial analysis, the concentration of the post digestion spike standard is equivalent to 2X the CRQL for all elements. If the matrix spike recovery for any element is outside control limits, the unspiked sample result for that element does not exceed four times the concentration of spike added and if the indigenous level in the unspiked sample for that element is higher than 2X the CRQL, then the post-digestion spike must be repeated with a spike concentration equivalent to 2X the indigenous level in the unspiked sample for those elements.

To prepare the preparation blank (PB), transfer 100mL of reagent water to a labeled digestion vessel. To prepare the LCS, transfer 1mL of the Intermediate ICV/Aqueous LCS standard to a labeled digestion vessel and dilute to 100mL with reagent water.

Add 2mL of 1:1 HNO₃ and 1mL of 1:1 HCl to each digestion vessel. Cover the digestion vessel(s) with a ribbed watch glass and place in a heated block digester maintained at a temperature of ~92-95°C. Heat until the sample volume is reduced between 25 and 50mL. Monitor the digestion to ensure that the sample does not boil and that the digestion vessel does not go dry. Cover with a watch glass to prevent additional evaporation and reflux for 30 minutes. After digestion is complete, cool the sample, adjust the volume to 100mL with reagent water and allow any insoluble material to settle.



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11.2 Analysis

Establish the operating parameters of the instrument following the manufacturer's instructions. Allow the instrument to equilibrate for at least 30 minutes prior to analysis. Check the sample flow through the pump tubing to ensure that flow to the nebulizer and drain flow is smooth. If the flow is erratic, check the tubing placement on the pump rollers and replace, as necessary.

Note: The drain flow must be smooth or instrument damage will occur!

Transfer approximately 50mL of ICP-MS Tune Working Standard Solution into an autosampler tube.

Transfer approximately 50mL of each working calibration standard, ICV, CRI, CCV, ICSA and ICSAB into an autosampler tube.

Transfer approximately 10 mL of each digestate into an autosampler tube.

Prepare the post-digestion spike by transferring 9.9mL of sample into an autosampler tube to which 0.1mL of post spiking solution has been added. Mix well.

Prepare the serial dilution by transferring 2mL of sample and 8mL of 2% nitric acid solution to an autosampler tube. Mix well.

On the PC connected to the instrument, select the autosampler template and enter the sample IDs in the order of analysis. Place the samples, serial dilution, post-digestion spike, calibration blank, mixed calibration standards, and performance check standards in the position on the autosampler rack that corresponds to their assigned position in the autosampler template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin analysis.

An example analytical sequence is given below:

Calibration Blank

Calibration Standard #1

Calibration Standard #2

Calibration Standard #3

ICV

ICB

CRI

ICSA

ICSAB

CCV

CCB

10 Samples*

CCV

CCB

9 Samples*

CRI
CCV
CCB

**The number of samples between each CCB/CCV (10) includes preparation blanks, laboratory control samples, matrix spikes, sample duplicates, serial dilutions and the post digestion spike.*

After analysis is complete, review the data against the criteria given in Section 9.0 for Quality Control and Section 10.0 for Calibration and Standardization. Perform corrective action, as needed. Dilute and reanalyze any samples that exceed the linear range and perform post digestion spikes as necessary.

Transfer the data to the laboratory NT server. After this occurs, transfer the data from the network server into the MARRS data processing software program. Route preparation log, analysis run log and associated raw data to the Inorganic Data Review department for secondary data assessment and report generation.

Note: The data processing software is configured to acquire three replicate integrations and uses the average of the three integrations for all QC and sample analyses for instrument calibration and quantification. The software also performs correction for internal standardization. A rinse blank is performed between each acquisition to flush the system and remove traces of the previous sample. The data system determines the concentration using the equations given in Section 12.0.

12.0 CALCULATIONS

12.1 Equation 1: Sample Concentration / Aqueous Sample

$$C_{(\mu\text{g/L})} = \mu\text{g/L}_{\text{DIG}} * (V_{\text{DIG}}/V_{\text{SAMP}})$$

Where:

$\mu\text{g/L}_{\text{DIG}}$ = ICP-MS Result

V_{DIG} = Digestate volume in mL

V_{SAMP} = Sample volume in mL

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

- 13.1 Primary review is performed and documented by the analyst that performed the digestion or analysis procedure. All data undergoes secondary review by a senior analyst or data review analyst. The review ensures that all data is reviewed against the performance criteria given in Section 9.0 for quality control and Section 10.0 for calibration and standardization. If the results are not within the established limits or criteria, corrective action is performed. If corrective action is not taken or is unsuccessful, the situation is documented and reported in the project narrative. All data that does not meet established criteria is flagged with the appropriate data qualifier and noted in the project narrative.



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14.0 METHOD PERFORMANCE

- 14.1 An Initial Demonstration of Capability is required for each analyst before unsupervised performance of this method.
- 14.2 An Initial Method Detection Limit (MDL) determination for each test method referenced in this SOP is performed following the procedure described in the reference method, 40CFR, Part 136, Appendix B and laboratory SOP LP-LB-009. The MDL is repeated annually or when a significant change to the method occurs. Significant changes include the use of alternate reagents or standard reference materials, new instrumentation or the use of alternate sample preparation procedures.
- 14.3 The upper limit of the linear calibration range is established quarterly for each element by determining the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. The linear calibration range used for the analysis of samples is determined from the resulting data. The upper linear dynamic range (LDR) limit is an observed signal no less than 10% below the level extrapolated from lower standards.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

- 15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 15.2 The following waste streams are produced when this method is carried out.
 - Acidic Waste – Satellite Container: 5 Gallon Polyethylene Container

Transfer the waste stream to the designated satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste*.

16.0 REVISION HISTORY

- 16.1 Title Page: Updated to current management team.
- 16.2 Section 6.0: Updated to current instrument model.
- 16.3 Section 14.0: Changed MDL frequency to annually and removed reference to IDLs.
- 16.4 Section 15.0: Added waste stream information.
- 16.5 Section 18.0: Added Table 2.0



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17.0 REFERENCES

- 17.1 Multi-Media, Multi-Concentration SOW for Inorganic Analytes, ILM05.3, January, 2004
USEPA Contract Laboratory Program.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

- 18.1 Table 1: *Analyte List, Contract Required Detection Limit, and Recommended Isotopes*
18.2 Table 2: Primary Materials Used
18.3 Table 3: *Internal Standard Component and Concentrations*
18.4 Appendix A: *Standard Preparation Tables*
18.5 Appendix B: *List of Definitions*



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Table 1: Target List, Contract Required Detection Limits & Recommended Isotopes

Element		CAS Number	CRQL ug/L	Isotope
Al	Aluminum	7429-90-5	--	27
Sb	Antimony	7440-36-0	2	121
As	Arsenic	7440-38-2	1	75
Ba	Barium	7440-39-3	10	137
Be	Beryllium	7440-41-7	1	9
Cd	Cadmium	7440-43-9	1	111
Ca	Calcium	7440-70-2	--	--
Cr	Chromium	7440-47-3	2	52
Co	Cobalt	7440-48-4	1	59
Cu	Copper	7440-50-8	5	63
Fe	Iron	7439-89-6	--	--
Pb	Lead	7439-92-1	1	208
Mg	Magnesium	7439-96-5	--	25
Mn	Manganese	7439-96-5	1	55
Ni	Nickel	7440-02-0	1	60
K	Potassium	7440-09-7	--	--
Se	Selenium	7782-49-2	5	82
Ag	Silver	7440-22-4	1	107
Na	Sodium	7440-23-5	--	--
Tl	Thallium	7440-28-0	1	205
V	Vanadium	7440-62-2	5	51
Zn	Zinc	7440-66-6	10	66

Table 2: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

1 – Always add acid to water to prevent violent reactions.

2 – Exposure limit refers to the OSHA regulatory exposure limit.



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Table 3: Internal Standard Solution Components and Concentration

Element	Concentration (ug/L)	Mass (amu)
Li	13000*	6 and 7
Y	50	89
Sc	100	45
Tb	25	159
Bi	55	209
In	50	115

**The concentration is set per the instrument manufacturer's recommendation.*

Appendix A: Standard Preparation Tables

ICP-MS Tune Standard Intermediate Solution

Single Element Stock Standard	Stock Standard Concentration (mg/L)	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
Li	10000	0.02	2000	1000
Be	1000	0.2	2000	1000
Mg	10000	0.02	2000	1000
Al	10000	0.02	2000	1000
Sc	1000	0.2	2000	1000
V	1000	0.2	2000	1000
Co	1000	0.2	2000	1000
Y	10000	0.02	2000	1000
In	1000	0.2	2000	1000
Ba	1000	0.2	2000	1000
Ce	1000	0.2	2000	1000
Tb	1000	0.2	2000	1000
Pb	1000	0.2	2000	1000
Th	1000	0.2	2000	1000
U	1000	0.2	2000	1000
Bi	1000	0.2	2000	1000

Solution: 2% Nitric Acid



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ICP-MS Tune Working Standard Solution

Single Element Stock Standard	Stock Standard Concentration (ug/L)	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
Li	1000	10	100	100
Be	1000	10	100	100
Mg	1000	10	100	100
Al	1000	10	100	100
Sc	1000	10	100	100
V	1000	10	100	100
Co	1000	10	100	100
Y	1000	10	100	100
In	1000	10	100	100
Ba	1000	10	100	100
Ce	1000	10	100	100
Tb	1000	10	100	100
Pb	1000	10	100	100
Th	1000	10	100	100
U	1000	10	100	100
Bi	1000	10	100	100

Solution: 2% Nitric Acid

Intermediate Calibration Standard Solution

Singe Element Stock Standard	Stock Standard Concentration (mg/L)	Initial Volume (mL)	Final Volume (mL)	Final Concentration (mg/L)
Al	10,000	2.0	500	40
Sb	1000	10	500	20
As	1000	1.0	500	2.0
Ba	1000	50	500	100
Be	1000	1.0	500	2.0
B	1000	10	500	20
Cd	1000	1.0	500	2.0
Ca	10,000	50	500	1000
Cr	1000	2.0	500	4.0
Co	1000	5.0	500	10
Cu	1000	10	500	20
Fe	10,000	10	500	200
Pb	1000	1.0	500	2.0
Mg	10,000	50	500	1000
Mn	1000	2.0	500	4.0
Mo	1000	10	500	20
Ni	1000	2.0	500	4.0
K	10,000	50	500	1000
Se	1000	1.0	500	2.0
Ag	1000	1.0	500	2.0
Na	10,000	50	500	1000
Tl	1000	1.0	500	2.0
V	1000	2.0	500	4.0
Zn	1000	10	500	20

Solution: 2% Nitric Acid

Working Calibration Standard Solutions

Element	Intermediate Calibration Standard (mg/L)	Calibration Level 3			Calibration Level 2 (Prepared from Cal 3)			Calibration Level 1 (Prepared from Cal 3)		
		Initial Volume (mL)	Final Volume (mL)	Final Concentration (mg/L)	Initial Volume (mL)	Final Volume (mL)	Final Concentration (mg/L)	Initial Volume (mL)	Final Volume (mL)	Final Concentration (mg/L)
Al	40	25	1000	1000	100	500	200	20	500	40
Sb	20	25	1000	500	100	500	100	20	500	20
As	2.0	25	1000	50	100	500	10	20	500	2.0
Ba	100	25	1000	2500	100	500	500	20	500	100
Be	2.0	25	1000	50	100	500	10	20	500	2.0
B	20	25	1000	500	100	500	100	20	500	20
Cd	2.0	25	1000	50	100	500	10	20	500	2.0
Ca	1000	25	1000	25000	100	500	5000	20	500	1000
Cr	4.0	25	1000	100	100	500	20	20	500	4.0
Co	10	25	1000	250	100	500	50	20	500	10
Cu	20	25	1000	500	100	500	100	20	500	20
Fe	200	25	1000	5000	100	500	1000	20	500	200
Pb	2.0	25	1000	50	100	500	10	20	500	2.0
Mg	1000	25	1000	25000	100	500	5000	20	500	1000
Mn	4.0	25	1000	100	100	500	20	20	500	4.0
Mo	20	25	1000	500	100	500	100	20	500	20
Ni	4.0	25	1000	100	100	500	20	20	500	4.0
K	1000	25	1000	25000	100	500	5000	20	500	1000
Se	2.0	25	1000	50	100	500	10	20	500	2.0
Ag	2.0	25	1000	50	100	500	10	20	500	2.0
Na	1000	25	1000	25000	100	500	5000	20	500	1000
Tl	2.0	25	1000	50	100	500	10	20	500	2.0
V	4.0	25	1000	100	100	500	20	20	500	4.0
Zn	20	25	1000	500	100	500	100	20	500	20

Solution: 2% Nitric Acid

Note: Working calibration standards Level 1 and Level 2 are secondary dilutions of calibration standard level 3, which is prepared from the intermediate calibration standard solution. The working calibration standards must be assigned an expiration date of 14 days from date of preparation or in accordance with the expiration date of the parent standard, whichever occurs first.



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CLP-MS-CRQL Stock Standard Solution

Stock Standard Element	Concentration (ug/mL)	CRI Working Standard Concentration (ug/L)
Ba Zn	10	10
Se Cu V	5	5
Sb Cr	2	2
As Be Cd Pb Ni Ag Ti Co Mn	1	1

CRI Working Standard Solution

Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)
CLP-MS CRQL Stock	1	1000

Solution: 2% Nitric Acid

6020ICS-0A Stock Standard Solution

Multi-Element Stock Standard	Stock Standard Concentration (ug/mL)	Prepared Standard Concentration (mg/L)
Cl	10000	1000
Ca Fe Na Al Mg P K S	1000	100
C	2000	200
Mo Ti	20	20

Interference Check (ICS) Solution / ICSA Working Standard

Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)
6020ICS-0A	10	100

Solution: 2% Nitric Acid

CLP-MS-ICS-B Intermediate Stock Standard Solution

Multi-Element Stock Standard	Stock Standard Concentration (ug/mL)	Prepared Standard Concentration (ug/L)
Al Sb As Ba Be Cd Cr Co Cu Pb Mg Mn Ni Se Ag Ti V Zn	2	20



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Interference Check (ICS) Solution / ICSAB Working Standard

Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)
6020ICS-0A	10	100
CLP-MS-ICS-B	1	100

Solution: 2% Nitric Acid

Intermediate ICV/Aqueous LCS Standard Solution

Single Element Stock Standard	Stock Standard Concentration (mg/L)	Initial Volume (mL)	Final Volume (mL)	Final Concentration (mg/L)
Al	10,000	1.0	200	50
Sb	1000	5.0	200	25
As	1000	0.5	200	2.5
Ba	1000	25	200	125
Be	1000	0.5	200	2.5
B	1000	5.0	200	2.5
Cd	1000	0.5	200	2.5
Ca	10,000	25	200	1250
Cr	1000	1.0	200	5.0
Co	1000	2.5	200	12.5
Cu	1000	5.0	200	25
Fe	10,000	5.0	200	250
Pb	1000	0.5	200	2.5
Mg	10,000	25	200	1250
Mn	1000	1.0	200	5.0
Mo	1000	5.0	200	25
Ni	1000	1.0	200	5.0
K	10,000	25	200	1250
Se	1000	0.5	200	2.5
Ag	1000	0.5	200	2.5
Na	10,000	25	200	1250
Tl	1000	0.5	200	2.5
V	1000	1.0	200	5.0
Zn	1000	5.0	200	25

Solution: 2% Nitric Acid



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Working ICV/Aqueous LCS Standard Solution

Element	ICV Intermediate Standard Concentration (mg/L)	Initial Volume (mL)	Final Volume (mL)	Final Concentration (ug/L)
Al	50	5	500	500
Sb	25	5	500	250
As	2.5	5	500	25
Ba	125	5	500	1250
Be	2.5	5	500	25
B	2.5	5	500	250
Cd	2.5	5	500	25
Ca	1250	5	500	1250
Cr	5.0	5	500	50
Co	12.5	5	500	125
Cu	25	5	500	250
Fe	250	5	500	2500
Pb	2.5	5	500	25
Mg	1250	5	500	1250
Mn	5.0	5	500	50
Mo	25	5	500	250
Ni	5.0	5	500	50
K	1250	5	500	12500
Se	2.5	5	500	25
Ag	2.5	5	500	25
Na	1250	5	500	12500
Tl	2.5	5	500	25
V	5.0	5	500	50
Zn	25	5	500	250

Solution: 2% Nitric Acid



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Matrix Spike Solution

CLPP-SPK-1 Stock Standard Solution

Multi-Element Stock Standard	Stock Standard Concentration (ug/mL)	Prepared Standard Concentration (ug/L)
Al Ba	2000	2000
Fe	1000	1000
Co Mn Ni V Zn	500	500
Cu	250	250
Cr	200	200
Ag Be	50	50

CLPP-SPK-5 Stock Standard Solution

Multi-Element Stock Standard	Stock Standard Concentration (ug/mL)	Prepared Standard Concentration (ug/L)
Sb	100	100
Cd Se Tl	50	50
As	40	40
Pb	20	20

Matrix Spike Working Standard Solution

Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)
CLPP-SPK-1	100	1000
CLPP-SPK-5	100	1000

Solution: 2% Nitric Acid



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Appendix B: List of Definitions

Accuracy: the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria.

Calibration: the establishment of an analytical curve based on the absorbance, emission intensity or other measured characteristic of known standard.

Calibration Blank (ICB/CCB): a volume of reagent water acidified with the same acid matrix as in the calibration standards.

Calibration Standards: a series of known standard solutions used to calibrate the instrument response with respect to analyte concentration.

Continuing Calibration Verification (CCV): a prepared standard solution used to verify the stability of the instrument calibration and instrument performance during the analysis of samples.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

ICP-MS-A technique for the multi-element determination of elements in solution. The basis of the technique is the detection of atomic ions produced by an ICP and sorted by mass/charge ratio.

Interference Check Solution (ICS): a solution of known concentrations of interfering elements that will demonstrate the magnitude of interference and provide an adequate test for any corrections.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): A prepared standard solution from a source separate from that of the calibration standards used to verify the concentration of the calibration standards and the adequacy of instrument calibration.

Instrument Detection Limit (IDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using



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a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which

the relative uncertainty is +100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Linear Dynamic Range: The concentration range over which the instrument response remains linear.

Matrix: the substrate of a test sample.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is +100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Percent Solids (%S): the proportion of solid in a soil sample.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Quality Control Sample: a control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Serial Dilution: the dilution of a sample by a factor of five.

Tune: the analysis of a solution containing a range of isotope masses to establish ICP-MS accuracy, resolution and precision prior to calibration.

ATTACHMENT 7

STL Burlington Particle-Size Distribution by Laser Light Scattering
(ASTM D4464)



STL

SOP No. LM-SL-D4464
Revision: 0
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Effective Date: 10/12/05
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**STANDARD OPERATING PROCEDURE
PARTICLE-SIZE DISTRIBUTION OF CATALYTIC MATERIAL BY LASER LIGHT
SCATTERING
ASTM D4464-00
Applicable Matrix: Soil**

APPROVAL SIGNATURES

Laboratory Director:

Christopher A. Ouellette
Christopher A. Ouellette

Date: October 7, 2005

QA Manager:

Kirstin L. McCracken
Kirstin L. McCracken

Date: October 7, 2005

Department Manager:

William S. Cicero
William S. Cicero

Date: October 7, 2005

Technical Director:
Geotechnical Testing

Matthew R. Duquette
Matthew R. Duquette

Date: October 7, 2005

Proprietary Information Statement:

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1.0 SCOPE AND APPLICATION

- 1.1. This SOP describes the laboratory procedure for the determination of particle size distribution in soil samples that contain sand, silt and clay.
- 1.2. Results obtained by this method or any other method for particle size determination utilizing different physical principles may disagree. The results are strongly influenced by physical principles employed by each method of particle size analysis. The result of any particle sizing method should be used only in a relative sense and should not be regarded as absolute when comparing results obtained by other methods.

2.0 SUMMARY OF METHOD

- 2.1. A prepared sample of particulate material is dispersed in water or a compatible organic liquid and is circulated through the path of a laser light beam or some other suitable source of light. The particles pass through the beam and scatter it. Photodetector arrays collect the scattered light, which is converted to electrical signals to be analyzed using Fraunhofer Diffraction, or Mie Scattering, or both. Scattering information, typically, is analyzed assuming a spherical geometry for the particles. Calculated particle sizes are, therefore, presented as equivalent spherical diameters.
- 2.2. This procedure is based on ASTM D4464-00.

3.0 DEFINITIONS

- 3.1. *Background-* extraneous scattering of light by material present in the dispersion fluid other than the particles to be measured. It includes scattering by contamination in the measurement path.
- 3.2. *Fraunhofer Diffraction-* the optical theory that describes the low-angle scattering of light by particles that are large compared to the wavelength of the incident light.
- 3.3. *Mie Scattering-* the complex electromagnetic theory that describes the scattering of light by spherical particles. It is usually applied to particles with diameters that are close to the wavelength of the incident light. The real and imaginary indices of light refraction of the particles are needed.
- 3.4. *Multiple Scattering-* the re-scattering of light by a particle in the path of light scattered by another particle. This usually occurs in heavy concentrations of a particle dispersion.

4.0 INTERFERENCES

- 4.1. Air bubbles entrained in the circulating fluid will scatter light and then be reported as particles. Circulating fluids typically do not require degassing, but should be bubble-free on visual inspections.

- 4.2. Contaminants, such as non-aqueous solvent, oil or other organic coatings on the sample may emulsify in an aqueous carrier, scatter light, and be reported as part of the particle size distribution. Samples containing such contaminants may be analyzed in a non-aqueous carrier solvent to dissolve the contaminants or washed free of the contaminant with a compatible aqueous solvent.
- 4.3. Reagglomeration or settling of particles during analysis will cause erroneous results. Dispersions shall be prepared so a stable dispersion is maintained throughout the analysis.
- 4.4. Insufficient sample loading (obscuration) may cause electrical noise interference and poor data reproducibility. High sample loading may cause excessive light attenuation and multiple scattering, resulting in erroneous particle size distributions.

5.0 SAFETY

- 5.1. Care should be taken to avoid exposure to the sample matrix since all environmental samples are potentially hazardous. Protective clothing, eye protection and disposable gloves should be worn when handling samples. All laboratory personnel must be familiar with the environmental health and safety plan described in the STL Chemical Safety Manual.

6.0 EQUIPMENT AND SUPPLIES

- 6.1. Malvern MasterSizer 2000 with Hydro 2000G wet-dispersion unit.
- 6.2. 40ml glass vials with caps
- 6.3. Disposable Pipets
- 6.4. Mechanical vortex machine

7.0 REAGENTS AND STANDARDS

- 7.1. Reagents
Deionized (DI) Water: Milli-Q System

8.0 SAMPLE HANDLING AND PRESERVATION

- 8.1. At least 50 grams of soil sample should be collected in glass or polyethylene jars. Immediately following collection the sample should be sealed and cooled to 4°C in order to preserve the moisture content of the sample.
- 8.2. Samples are stored from the time of receipt in the laboratory until 30 days after delivery of the reconciled data package report. Unless otherwise specified by a federal, state or

client-specific protocol, samples are disposed of after 30 days in a manner that complies with all applicable regulations.

9.0 QUALITY CONTROL

Not Applicable

10.0 CALIBRATION AND STANDARDIZATION

- 10.1. Calibration of the instrument is performed yearly by the manufacturer. Scheduling of calibration is arranged by the department supervisor.

11.0 PROCEDURE

11.1. Instrument Preparation

Allow the instrument to warm up for at least 30 minutes before performing sample analysis.

Ensure that the dispersion unit is clean and filled with DI water.

11.2. Sample Preparation

Label a clean 40ml vial with the sample #.

Mix the sample in the sample container, making sure to completely homogenize the sample.

Add approximately 10 grams of sample to the labeled 40ml vial.

Add DI water to the sample, filling the vial approximately 2/3 full.

Cap the vial, and using the mechanical vortex machine disperse the sample inside the vial. Visually inspect the sample, ensuring that any agglomerations have been dispersed.

11.3. Particle Size Test

Open the Mastersizer program on the computer terminal.

Select "Run Existing SOP" on the menu, and select "STL-BTV-D4464" from the SOP list.

Save the data file using the SDG # as the file name.

Follow the on-screen instructions to perform sample analysis.

After each sample is run, select "Run this SOP again" from the menu. Continue selecting this option until finished analyzing samples.

When finished analyzing samples, select "Measure" and in the measurement window, open the "Accessories" tab. Briefly raise the pump RPM to 2000, then stop the pump and close the "Accessories" tab. Close the measurement window.

After final analysis, ensure that the dispersion unit and cell are filled with clean DI water. Turn off the particle size analyzer.

12.0 CALCULATIONS

Not Applicable

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

- 13.0. Document any problems encountered during sample analysis so they may be properly addressed in the project narrative. Perform primary and secondary data review following the guidance given in laboratory SOP LP-LB-003 *Data Review*.

14.0 METHOD PERFORMANCE

Not Applicable

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

- 15.1. The laboratory optimizes technology to minimize pollution and reduce the production of hazardous waste whenever possible.
- 15.2. The laboratory procedures for waste management comply with applicable federal, state and local regulations and are described in SOP LP-LB-001HAZWD.

16.0 REFERENCES

- 16.1. Standard Test Method for Particle Size Distribution of Catalytic Material by Laser Light Scattering, ASTM D4464-00, Volume 04.08 Soil and Rock, American Society for Testing and Materials, Philadelphia, Pa., 2000.

17.0 REVISION HISTORY

- 17.1 This is the first version of this SOP.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

Not Applicable